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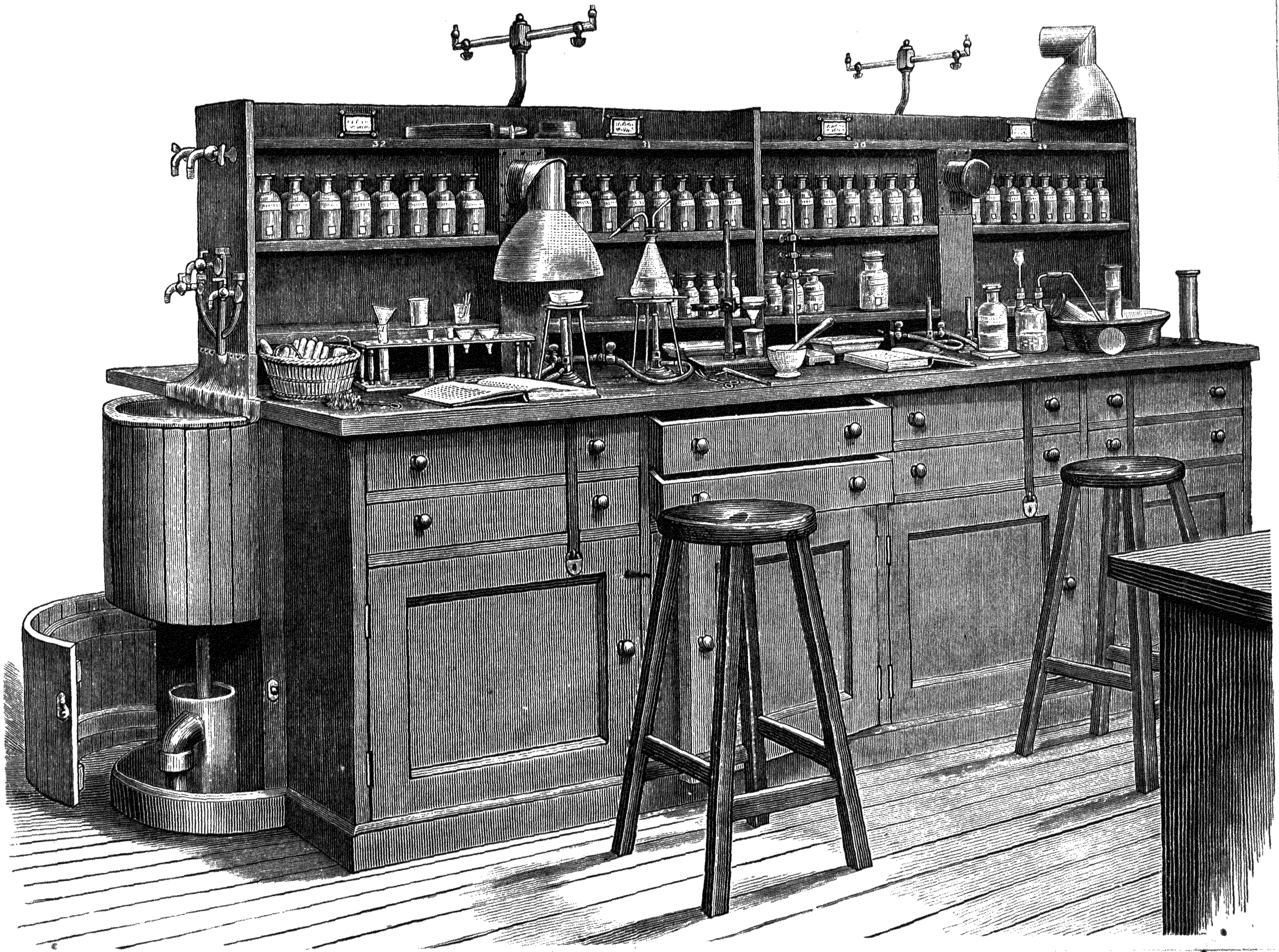

Frontispiece.] [*See page* 441.

LABORATORY BENCH, UNIVERSITY COLLEGE, NOTTINGHAM.

A TREATISE
ON
QUALITATIVE ANALYSIS
AND
PRACTICAL CHEMISTRY

ADAPTED FOR USE IN THE LABORATORIES OF
COLLEGES AND SCHOOLS

BY

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PREFACE.

THIS Treatise was commenced to supply a course of Practical Chemistry to my own classes. I was encouraged to proceed with it by finding that the want of a sufficiently systematic and explanatory Laboratory Text-Book was very widely felt.

It has been my aim throughout to give all necessary directions so fully and simply as to reduce to a minimum the assistance required from a teacher. The language employed has, I trust, been rendered simple and intelligible, by avoiding the unnecessary use of scientific terms, and by explaining or paraphrasing in ordinary words any such terms when they are introduced for the first time.

The directions for working, and the descriptions of the preparation and use of apparatus, have been given more fully than is usual. My own experience, which is confirmed by that of other teachers, convinces me that one of the most serious hindrances to the utility of many of the smaller Text-Books on Practical Chemistry is the conciseness of the language employed; on this account it is unintelligible to the student unless it is supplemented by very copious verbal explanation from the teacher.

Whilst the very desirable amplifications above referred to have been made, all such instruction as does not directly bear on the ordinary requirements of the laboratory student have been omitted, and the supplementary and explanatory portions have been inserted in smaller type.

I have also thought it best to avoid entering into any lengthy theoretical explanations. The modern teaching of chemistry is in practice very appropriately divided into two departments; namely, theoretical instruction imparted by classes and lectures, or by the study of text-books of theoretical chemistry; and practical experience acquired in a chemical laboratory by working according to the directions of a Practical Text-Book. Since by this system the student has time and opportunity afforded him elsewhere for the study of the theoretical and descriptive portions of the science, it is unnecessary and undesirable that his Practical Text-Book should tempt him to bestow valuable time in the laboratory upon the consideration of matters which do not directly bear upon his experimental work.

The analytical reactions and methods have been carefully worked through from the text by myself and by the members of my classes: the accuracy and intelligibility of their description have thus, I hope, been secured. Only those reactions and methods, which are commonly employed for analytical purposes, have been described. In selecting analytical methods, I have felt it desirable that those chosen should be as simple and easy of execution as possible. In some cases, however, the methods which are most eligible on these grounds have proved on trial to be unsatisfactory in accuracy and delicacy. They have therefore been abandoned in the Analytical Tables, and other methods, which are recommended by their trustworthiness rather than by their simplicity, have been entered. In such cases, however, I have described the more simple methods also, since they may be employed in analyses in which minute quantities of substances have not to be tested for.

I have felt it advisable to introduce reactions which are useful in general analysis rather than those which claim to be merely interesting and instructive; and I have not hesitated to adopt methods which are not quantitative, if they are easy and accurate for qualitative analysis.

The book is intended to give a course of instruction, which is suited to the general requirements of laboratory students. Those who are using the book for any special purpose, or who wish to prepare themselves for any particular examination, should note that it has been divided into seven Sections. The objects of these Sections are stated below and their Contents are given in detail on pages xi–xxii.

The first six Sections, omitting the Fifth, contain a good practical course for senior students. This course may, however, be somewhat modified and amplified to suit junior students, who will also possibly substitute the Fifth Section for the Sixth.

The Fifth Section has indeed been inserted for the use of medical, pharmaceutical and engineering students, and for those who are preparing for practical examinations in which proficiency in the analysis of a simple salt only is required of the candidate. This is the standard fixed for some of the medical examinations and for some of the more elementary examinations in analytical chemistry.

The Sixth Section supplies a full set of Tables, which are suited for the analytical work of advanced students, and will be found sufficient to qualify a student for the higher University examinations and for the examination of the Institute of Chemistry in qualitative analytical chemistry.

The Seventh Section of the book contains suggestions regarding the fitting and furnishing of the laboratory, together with full lists of all the apparatus, reagents and chemicals which are required in working through the different Sections of the book. There is added a list of general apparatus, with a description when necessary of its construction and use.

In this Section there will also be found full and systematic descriptions of the most simple methods for preparing the different solutions which are required in analysis, with a statement of the strength which is most appropriate for each. Experience has proved that this information is not only very convenient, but tends largely to secure economy of time and money in the preparation for laboratory work.

This Section concludes with Tables of Atomic Weights, and of French and English Weights and Measures.

Symbolic notation has been employed throughout the Sections on analytical chemistry. In its most concise form, this chemical shorthand conduces so much to brevity in writing down results, that no other plea is required for its use. The simple plan of entering upon the label of each bottle in the laboratory not only the chemical name but also the chemical formula of its contents, will prevent the general use of chemical formulæ from causing perplexity to beginners. A reference to the lists of chemicals in Section VII. will also furnish the formula which corresponds to the name of any substance. It has, however, been thought well to insert the chemical name of each substance together with its formula, when the substance is first referred to in the text.

The only point in nomenclature worthy of note is that the hydroxides of the metals are frequently referred to by their less modern designation of hydrates.

The arrangement of all Tables *across* instead of *along* the pages renders the turning of the book unnecessary, a convenience which will be appreciated by all laboratory students.

The "Tables of Differences" contain a summary of the differences of behaviour of the members of each Analytical

Group with reagents. These Tables serve as useful guides to elementary analytical work, and furnish explanations of the Analytical Tables.

It is almost superfluous to mention that free use has been made of the older standard treatises on Chemical Analysis; and my work has partly consisted in remodelling and combining the information, which they furnish, in a systematic and convenient form. My acknowledgments are also due to many contemporary experienced teachers who have made valuable suggestions of addition and emendation.

The book is intended to furnish a course of general laboratory instruction in practical chemistry and qualitative analysis. Such a course should precede the higher training of the professional and pharmaceutical chemist and of the medical man, and the more special training of the technical chemist and of the chemical engineer. Sound elementary instruction in practical science is now, however, generally valued not only as a preparation for a professional career, and for many branches of manufacturing industry and enterprise, but also as an important branch of general mental training.

The present Edition has undergone revision mainly in Section II. on the preparation and detection of gases; and in Section IV., Part IV., and the latter part of Section VI., which deal with the reactions and detection of organic substances.

I have again to thank Professor Coleman, A.R.C.Sc., F.I.C., for suggestions in connection with this revision.

F. C.

LONDON, 1908.

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SECTION VII.

Laboratory Fittings, Apparatus, Chemicals, and Reagents.

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APPARATUS FOR EACH BENCH-LOCKER.

1	Bunsen's-burner with tubing (**1**).
1	Test-tube stand, with twelve holes.
12	Test-tubes.
1	Test-tube brush (**13**).
2	Boiling-tubes.
2	Round glass plates.
2	Porcelain dishes.
2	Watch-glasses.
1	Four-ounce conical flask.
1	Pestle and mortar.
1	Iron tripod-stand.
1	Piece of coarse iron wire-gauze.
3	Glass funnels, and some cut filter-papers.
3	Small beakers.
3	Glass rods (**10**).
1	Piece of platinum-foil.
2	Pieces of platinum-wire (**9**).
1	Mouth-blowpipe (**6**).
1	Pipe-clay triangle (fig. 81, **1076**).
1	Wash-bottle (**12**).
1	Retort-stand.
1	Wooden filter-stand (fig. 49, **92**).
1	Pair of brass crucible tongs.
1	Small horn or vulcanite spatula.
1	Wicker draining-basket (**15**).

A fuller description of this apparatus will be found in paragraph 1121. Most of the apparatus may be seen on the bench in the Frontispiece illustration. The reference numbers in brackets refer to paragraphs in which the apparatus is described.

SECTION I.

PREPARATION AND USE OF APPARATUS.

General Remarks.—A student who is commencing work in a general laboratory will find on the preceding page a list of the apparatus which should be contained in his private locker. This apparatus should be looked through, fitted, and cleansed, as is directed in this Section.

In the Seventh Section will be found particulars of other apparatus, which may be used by many students in common; also lists of the various reagents, and the methods of preparing them.

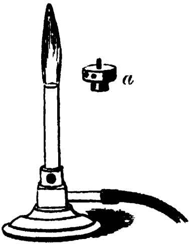
For convenience in reference, paragraph numbers are printed in the text in black type, and in square brackets at the top of each page.

Paragraphs which are printed in small type may be passed over if the student's time is limited.

FLAMES FOR HEATING.

1. The Bunsen Burner (fig. 1) is generally employed in the laboratory for heating purposes. It is so constructed that coal-gas, entering near its base, is mixed with a proper proportion of air before it is burnt. The air is drawn in through holes at the lower part of the burner, and the mixture of gas and air is burnt at the top of the upright tube. The oxygen of the air, which is thus mixed with the gas, burns the carbon in the body of the flame. Accordingly the luminosity, which depends upon the existence in the flame of unburnt carbon or carbon compounds, is destroyed.

FIG. 1.



BUNSEN-BURNER.

The Bunsen-flame is therefore mainly useful, because it deposits no particles of soot when it is in contact with any surface. Its high temperature and non-luminosity also render it very valuable for producing flame-colorations.

The burner should be provided with some means for partly or entirely closing the air-holes when requisite. This is usually effected by turning round a loose perforated ring outside the holes. If the flame is reduced to small dimensions, the supply of air should be partly shut off, else the flame is apt to recede.

When the burner is to be used, it is connected by means of a piece of tightly-fitting rubber tubing, about 5-16ths of an inch in internal diameter, with the tube which supplies gas to the working-bench. The tap is then turned on, and in a few seconds the gas is lighted. The flame should be almost perfectly colourless and non-luminous.

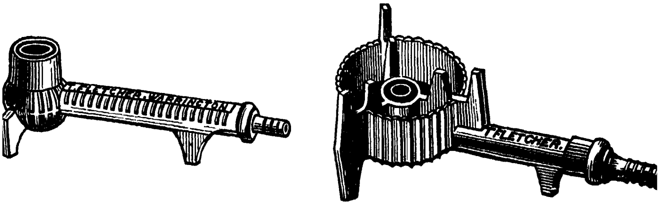
Occasionally the gas will *burn below*, that is, at the bottom instead of at the top of the burner. This usually happens when the flame is small and the quantity of air admitted through the holes is too great. It also occurs when the burner is lighted too soon after turning on the gas-tap. A luminous flame may then be seen through the holes near the base of the burner, burning from the small gas-jet inside. The flame at the top of the burner will also be long and somewhat luminous, and will emit a very unpleasant smell, which is due to the incomplete combustion of the gas. In such a case the flame should be at once extinguished by pinching the rubber tubing close to the burner, and the gas should be relighted after it has escaped for a few seconds.

The above statements should be proved by trial, and the effect of partially or entirely closing the air-holes of the burner should be observed.

The Rose-top (fig. 1 *a*) is employed for diffusing the heat of the flame over a large surface. It is a small perforated metal cap which is placed upon the top of the burner, and yields a circle of flames. The rose-top is put on the burner and removed from it by means of crucible-tongs. It is of course very hot after being used, and should never be handled, or placed upon wood or glass, until it is cool.

2. **Fletcher's Solid-flame Burner** is most convenient and satisfactory for heating large vessels. In its smallest size (figs. 2, 3) this burner is also very convenient on the student's working-bench. The burner shown in the figures is the Bunsen-Argand. It furnishes a short, compact, and hot flame, and it presents the advantage that the air-supply is self-adjusting. The form of this burner, shown in fig. 3, is furnished with supports for flasks and dishes which are being heated.

FIG. 2. FIG. 3.

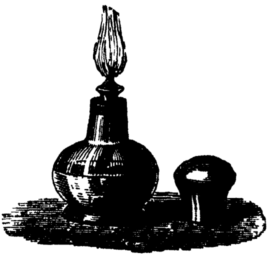


FLETCHER'S BURNERS

3. **The Spirit Lamp** is occasionally employed instead of a gas-burner; but for general purposes it should only replace the gas-burner when coal-gas cannot be obtained.

The spirit-lamp (fig. 4) consists of a glass vessel containing methylated spirit, into which dips a cotton wick supported by means of a brass or stoneware wick-holder. When the lamp is not in use the upper end of the wick should be always covered with the glass cap, in order to prevent evaporation of the spirit. If the spirit is tolerably free from resinous matter, its flame will be non-luminous and will deposit no soot. In this lamp the spirit is vaporised as it comes into contact with the flame.

FIG. 4.



SPIRIT-LAMP.

Other Special Burners for consuming a mixture of air with vaporised methylated spirit or petroleum, or with acetylene gas, are now obtainable, and may be used in laboratories where a supply of coal-gas is not available.

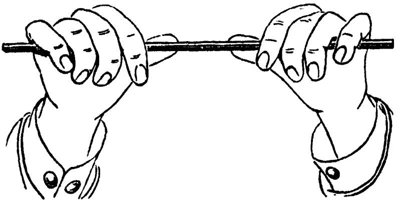
A burner similar in form to the Bunsen-burner (1) for burning a mixture of vaporised methylated spirit with air, and a metal lamp which furnishes a smokeless, non-luminous flame with vaporised petroleum oil, are described in paragraph 1119. A modified Bunsen-burner, suitable for burning acetylene, may now be purchased; and since acetylene can be prepared with great ease from calcium carbide. and then stored in a small gas-holder, such burner can be used in laboratories which are situated at a distance from gas-works, and an ordinary laboratory attendant may readily perform the duties of gas-maker

CUTTING AND BENDING GLASS.

4. **Glass Tube or Rod is Cut** by laying it upon a flat surface, and making a deep scratch with the edge of a three-cornered file at the point to be cut. The glass is then held with both hands, one on each side of the scratch and close to it, and a gentle pressure is exerted upon the glass as if trying to break it across (fig. 5). If the file-mark has been made sufficiently deep, the glass will readily break at this point.

The sharp edges of a freshly-cut rod or tube should always be at once rounded. This is effected either by holding the end of the tube in the Bunsen-flame or blowpipe-flame until the edges are partly melted (fig. 8, p. 6), or by rubbing off the sharp edge with the face of a file.

FIG 5.



CUTTING THE GLASS.

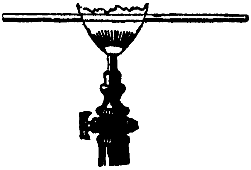
Select a length of hard-glass tubing, which is about one-eighth of an inch in internal diameter. Cut from it one piece about ten inches and two pieces about five inches long, and carefully round off their sharp freshly-cut edges. Also cut off three pieces of glass rod, respectively seven, six, and three inches in length. Keep these for future use.

5. **Glass Tube is Bent** by holding it in the upper edge of a common fish-tail gas-flame, so as to heat at least two inches of the glass (fig. 6). The tube is supported by both hands, one on either side of the flame; and is constantly turned slowly round on its axis, in order to heat all sides

equally. As soon as the glass is felt to be soft and pliable, it is taken out of the flame and is at once quickly bent to the required angle. The heated part must not be allowed to touch anything until it is cold. The soot is then removed from it by a cloth or by a piece of paper.

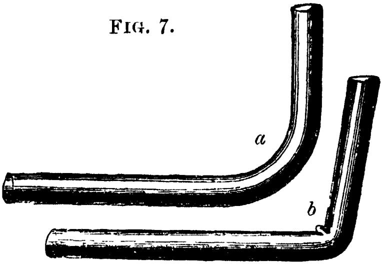
If a bend is properly made, it should be a curve and should not alter the bore of the tube (fig. 7 *a*). If a sharp angle is made, the bore will be narrowed (fig. 7 *b*), and the bend will be unsightly and weak, and liable to break under a small strain.

FIG. 6.



BENDING GLASS.

FIG. 7.



GOOD BEND (*a*), BAD BEND (*b*).

Bend the longest piece of glass tubing (4) at right angles, so that the shorter part is about two inches in length. Keep this for future use.

The Bunsen-flame is not suitable for bending ordinary glass tubing ; its shape is inconvenient, and the flame is so hot that it softens the glass too much and causes it to get out of shape.

Glass rod, hard glass tube, and tube with thick walls or small bore, however, may be bent in the Bunsen-flame, or even in the blowpipe-flame.

THE BLOWPIPE, AND ITS USE.

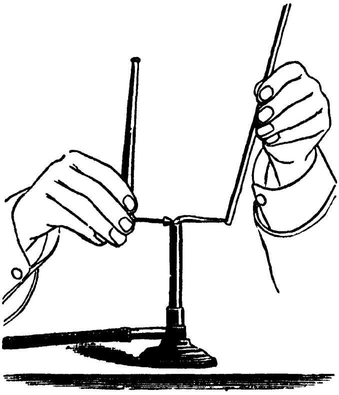
6. **The Blowpipe** is used for producing a small but very hot flame, which can be made to assume any desired direction. This is effected by blowing a fine stream of air from the mouth through a gas-flame. The blowpipe is held in the right hand, with its finely pierced tip resting on the

edge of the burner, and just inside the flame (fig. 8). The mouthpiece is then taken between the lips; and after the cheeks have been blown out to their full extent, the air contained in them is forced out through the jet. This produces a small pointed conical flame in the direction of the blast.

The chief difficulty in using the blowpipe properly is experienced in maintaining the blast of air uninterrupted by the breathing. Patient trial will, however, remove this difficulty.

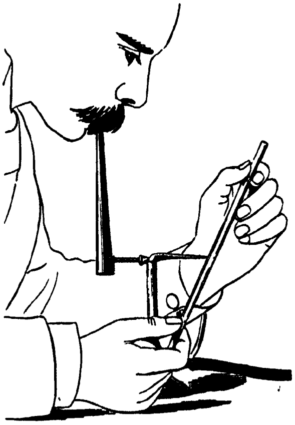
It must be borne in mind that the cheeks are to be kept constantly inflated with air, and that the air must be forced through the blowpipe *by the pressure of the cheeks, and not by the action of the lungs.* Breathing is carried on through the nose; and the mouth is occasionally replenished with air from the throat, the blast being meanwhile maintained by the cheek-muscles only.

FIG. 8.



ROUNDING END OF GLASS ROD.

FIG. 9.



DRAWING OFF GLASS TUBE.

It is frequently necessary to have both hands free while the blowpipe is being used. This may be secured by resting the blowpipe-jet upon the top of the gas-burner and supporting the mouthpiece by the lips alone, as is shown in fig. 9.

A small flame, made luminous by nearly closing the air-holes of the Bunsen-burner, is better suited for most blow-pipe-work than the ordinary non-luminous flame.

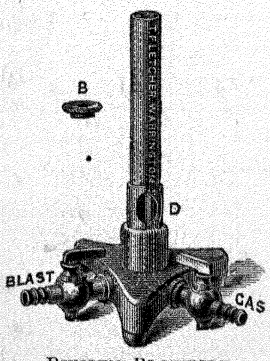
It is important that the hole in the nozzle of the blow-pipe should be small, else a good flame will not be obtained, and it will also be found difficult to maintain a continuous blast.

The student should, as soon as possible, acquire dexterity in the use of the mouth blow-pipe, since its flame serves for many of the shorter heating processes of the chemical laboratory.

7. **A Fletcher-Bunsen blowpipe** (fig. 10) which is fed with air from the Fletcher foot-bellows (fig. 11), or from a mechanical or water blower, is often advantageously employed for maintaining a high temperature in fusion processes, or for extensive glass-working or glass-blowing. It will also be found convenient for many other purposes.

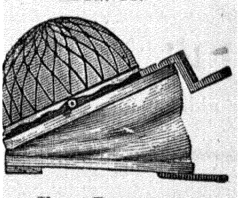
This special form of Bunsen-burner blowpipe (fig. 10) is suitable for many purposes. It consists of a somewhat large Bunsen-burner (I), which is furnished with two taps. One of these taps controls the gas-supply, the other controls the air-blast from the bellows.

FIG. 10.



BUNSEN BLOWPIPE.

FIG. 11.



FOOT-BELLOWS.

When a Bunsen-flame is required, the gas is supplied to the burner, and the air-supply is adjusted at the base of the burner by the ring D.

When the blowpipe-flame is to be used, the metal ring B is placed upon the top of the burner so as to reduce the aperture, and the supply of gas and the air-blast are regulated until a flame of the necessary character is obtained.

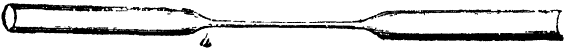
The Bunsen Gas-blowpipe (1118) will be found better adapted than the Fletcher-blowpipe for more elaborate glass-blowing and working.

The Herapath Gas-blowpipe (1118) is convenient for smaller heating purposes. It can, if desired, be readily blown by the mouth. It may be purchased without the foot and the tap, the upright tube being then slipped down the tube of a Bunsen-burner.

A Petroleum, or Spirit, Blowpipe (1119) may be used where coal-gas is not available. It possesses the advantage of being portable and of requiring no bellows or air-blast.

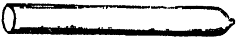
8. Small Ignition-tubes.—One of the pieces of hard-glass tubing, about five inches in length (4), is drawn out at its middle point by heating it strongly in the blowpipe-flame (fig. 9). While the tube is being heated, it is held in both hands, and is constantly turned round upon its long axis in order to secure uniform heating. As soon as the glass is softened, it is gradually drawn out by pulling the ends of the tube in opposite directions. The narrowed portion of the tube (fig. 12) is then cut across (4) at its middle point. By heating the conical part (*a*) in the blowpipe-flame, the narrow tube may then be drawn off, and a small closed tube is obtained (fig. 13). If the closed end is heated to redness in the blowpipe-flame, it may be expanded into a small bulb by gently blowing down the tube.

FIG. 12.



GLASS TUBE DRAWN OUT.

FIG. 13.



IGNITION-TUBE.

Small test-tubes, three inches long by half an inch in diameter, will also serve for ignition-tubes.

9. Mounted Platinum wires.—Select a length of platinum wire about as stout as an ordinary pin. Ascertain that the wire does not permanently impart colour to a Bunsen-flame when it is heated to bright redness in the flame. Cut off two pieces of this wire, each about two inches in length, and proceed to fix them in glass handles in the following way, in order to prevent them from being lost and to facilitate their being held in the fingers.

Draw out at its middle point (fig. 12) the piece of glass tube (4) about five inches in length, and cut it across at the middle of the narrow portion. Each of the pieces of tube thus obtained serves for the handle of a wire.

Break off the narrow part of the tube until it extends only about a quarter of an inch from the shoulder, *a* (fig. 12). Insert the end of the platinum wire into this narrow opening. Then hold the end of the tube, containing the wire, in the blow-pipe flame, until the glass melts and thickens around the end of the wire, fixing it firmly when cold (fig. 14). The free end of the wire may then be rolled round a stout wire into a loop about an eighth of an inch across.

FIG. 14.

MOUNTED PLATINUM WIRE.

10. Glass Stirring-rods.—Remove by a file any small projections from the ends of each of the glass-rods, which were made (4) by cutting some solid glass-rod into lengths of about three, six, and seven inches. Then heat both ends of each rod to redness in the tip of the blowpipe-flame, the rod being meanwhile constantly turned round on its long axis (fig. 8, p. 6). The sharp edges of the glass are thus rounded by fusion. The end of the rod must not be allowed to touch anything until it is cool.

Sometimes a very slender glass rod is required. This may be obtained by heating a suitable point in an ordinary rod with constant rotation in the blowpipe-flame until the glass is well softened, and then drawing the glass out to the requisite fineness. See fig. 9 (p. 6) and fig. 12.

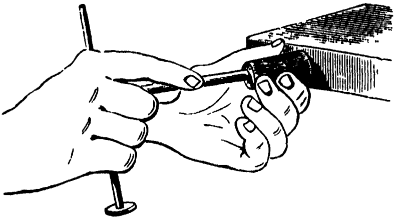
Boring Corks.

11. A Cork may be Bored, for the insertion of a glass tube, by gradually pushing through it a sharply pointed and slender round file with a constant twisting movement, and then enlarging the hole with the file to the necessary size.

The Cork-borer, which is a brass tube sharpened at one end, is, however, more commonly used.

A borer must be selected which is rather less in size than the glass tube which is to be inserted into the cork. The cork is then pressed against a wooden surface or grasped firmly in the hand, and the perforation is made by gently pushing the borer through it with a constant movement of rotation backwards and forwards upon its axis (fig. 15). Caution and practice will enable the student to make a clean straight hole without damaging the surrounding parts of the cork.

Fig. 15.



Boring a Cork

In making a single central hole through a cork, it is well to bore from the centre of one end of the cork halfway towards the other end, and then reverse the cork and bore a hole to meet this from the centre of the opposite end. In this way a hole which is central in position throughout the cork is most easily obtained.

A slender round file is used for smoothing the sides of a hole which has been made by a cork-borer, or for slightly enlarging it. Great care must be taken to leave the hole round in shape, and not to enlarge it so much that the glass tube fits loosely.

The cutting edge of the cork-borer is sharpened by rubbing its inner surface with a round file, and then rubbing the outer surface obliquely with the face of a flat or triangular file.

Rubber Stoppers are perforated in a similar manner by a sharp borer which is either partly filled with water, or is moistened with alcohol or glycerine.

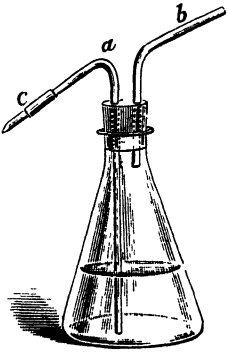
FITTING THE WASH-BOTTLE.

12. The Wash-bottle.—A thin, flat-bottomed, conical flask, about eighteen ounces in capacity, and not less than an inch in diameter in the neck, should be fitted as is shown in fig. 16.

For this purpose a sound cork is selected which is slightly too large to enter the neck of the flask. The cork is softened by rolling it backwards and forwards under the foot with gentle pressure; it must now fit tightly into the neck of the flask.

Two pieces of glass tubing, rather longer than the tubes (*a*, *b*), are then bent (5) into the form shown in fig. 16. Their ends are cut off to the right length, and the sharp edges are rounded by holding them in the Bunsen-flame, or by rubbing them with the face of the file.

FIG 16.



THE WASH-BOTTLE.

Two parallel holes are then bored through the cork, with a proper sized cork-borer (11) or by means of a round file. The holes must be somewhat smaller than the glass tubes, and must not run into one another or to the outside of the cork. They are smoothed, and slightly enlarged if necessary, by means of the round file.

Into these holes the tubes (*a*, *b*) are then gently pushed with a twisting motion. They must enter somewhat stiffly, but without requiring much pressure. If the holes have been carelessly made too large, the tubes may often be made to fit by slipping upon them little pieces of wetted narrow india-rubber tubing, or by putting the wetted pieces of india-rubber tubing into the holes in the cork before the glass tubes are pushed in.

A Rubber Stopper is much more durable than a cork for this and for most other chemical purposes. It may be purchased with two holes already made, or may be perforated by a sharp well-wetted cork-borer (11), or by a wetted round file. Both the glass tubes and the inside of the holes should be well wetted before the tubes are pushed in, since water serves as a lubricant for glass against india-rubber.

Before the further fitting of the wash-bottle is proceeded with, the cork with the tubes is inserted into the neck of the flask. One tube is then closed with the finger, and air is blown through the other tube into the flask, so as to produce pressure in its interior. The cork is meanwhile carefully watched in order to ascertain whether there is any escape of air. A leakage is as a rule easily heard : but if the outside of the cork is wetted with water, any escape of air-bubbles will be readily seen.

If the cork is air-tight, a piece of rubber tubing, about an inch in length, is pushed upon the upper end of the tube (*a*). A short jet (*c*), which has been made by drawing out a piece of glass tubing in the flame (figs. 9, 12, pp. 6, 8), is fitted into the other end of the rubber tube. The narrow opening of the glass jet may be contracted, if necessary, by holding it in the flame for a short time and slightly fusing the edges of the glass.

If the wash-bottle is to be heated, the neck of the flask should now be bound round with twine like the handle of a cricket bat, or tightly covered with a folded strip of flannel. The neck, when thus protected, can be grasped with comfort even after water has been boiled in the flask and the neck has become heated by steam.

The wash-bottle is now filled about two-thirds with distilled water, and is ready for use.

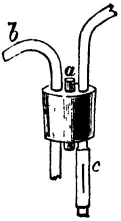
Tap-water should never be kept in the wash-bottle.

A fine stream of water may be obtained from the jet (*c*), by blowing down the tube (*b*). This stream serves for washing precipitates and for other purposes. A larger stream may be obtained by inverting the flask, when the water will flow out from the end of the tube (*b*), air entering meanwhile by the tube (*a*): this stream can be made to issue with greater force and speed by blowing down the tube (*a*).

When hot water is required, the wash-bottle is supported on a tripod-stand upon a piece of coarse iron-wire gauze, and is heated by the Bunsen-flame: or it may be heated by the small Fletcher-burner (fig. 3, p. 3).

When Boiling Water, or a Liquid which gives off Vapour or Gas, is used in the wash-bottle, a special stopper, shown in fig. 17, should be fitted into the neck of the bottle, in order to prevent the vapour of gas from entering the mouth.

FIG 17.



FITTING FOR HOT-WATER BOTTLE.

The stopper is perforated with three holes. Through the centre hole is pushed a short glass tube (*a*), which terminates just above and just below the stopper.

The other holes carry the jet-tube (*b*) and the blow-tube (*c*), as in the ordinary wash-bottle. The lower end of the blow-tube, however, is fitted with a valve, which is formed by slipping over it a piece of rubber tube with a slit cut in it (*c*) and closed below by a piece of glass rod.

While the wash-bottle is being used the tube (*a*) is closed with the finger, and air is blown in through the valve (*c*). The valve prevents air from returning through the blow-tube and introducing vapour or gas into the mouth. When the stream is to be stopped, the finger is removed from the tube (*a*) and the pressure of air in the interior of the flask is thus released.

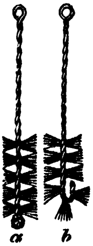
CLEANING APPARATUS.

It is indispensable that all glass and porcelain apparatus should be kept scrupulously clean, and before the student begins his work, he will do well to clean his set of apparatus as is directed below.

13. **Test-tube Brush.**—This brush is constantly in use for

cleaning glass and porcelain apparatus. The piece of sponge, which is ordinarily fastened on the end of the brush (fig. 18*a*), does not adapt itself well to the bottom of test-tubes and boiling-tubes. A much more efficient end is given to the brush by removing the sponge and then bending back the end of the wire stem sharply upon itself at a point just above where the hairs commence (fig. 18 *b*). By slightly curving the part of the stem carrying hairs, the brush will better adapt itself to curved surfaces such as those of porcelain dishes.

FIG. 18.



TEST-TUBE CLEANER.

14. Test-tubes, Beakers, Funnels and Porcelain-dishes are washed in a stream of tap-water, their surfaces being rubbed meanwhile by the test-tube cleaner (fig. 18 *b*). The brush must be cautiously moved as it approaches the bottom of a test-tube or boiling-tube, since the glass is very thin, and is therefore easily broken by undue pressure.

Glass funnels should have their stems cut off to within about an inch from the shoulder (4), and the sharp outer edge thus produced should be at once removed by rubbing it with the face of a triangular file. The inside of the shortened neck can then be easily cleaned by rubbing it with a strip of wood; or by inserting an ordinary tobacco-pipe cleaner from the shoulder of the funnel so as not to cut the hairs.

Occasionally the cleaning brush fails to remove strongly adhering stains. Hot dilute hydrochloric acid will remove most such stains; but it is sometimes necessary to heat a little strong sulphuric or nitric acid in a vessel in order to cleanse it. Hot solution of caustic potash or of ammonia may also often be used with advantage to remove grease. In fact, when a substance is being removed from a vessel to which it strongly adheres, it is well to consider what the substance is, and then remove it by a liquid in which it is easily soluble.

Apparatus should be washed as soon as possible after it has been used, since it is usually more difficult to cleanse the surface after the apparatus has stood aside for some time.

Each article, after it has been carefully washed, and then repeatedly rinsed out with tap-water, should be placed upside down in the small wicker-basket to drain. If it is required for immediate use for analysis, it should be rinsed out with a little distilled water. The removal by these means of washing-water is necessary, because tap-water usually contains dissolved impurities.

The interior of flasks and bottles is often most conveniently cleansed from adherent matter mechanically by shaking water and sand round in the interior. Sawdust or shreds of paper may replace the sand.

15. **Draining-Basket.**—Before apparatus is placed in the locker, it should be made a rule to wash all glass and porcelain which is not in actual use, and then to place it in a wicker basket to drain. The basket is then put away with its contents. Dirty apparatus should never be kept in the basket.

All iron apparatus should be carefully dried, and must be kept in a dry place to prevent it from rusting. *Metal apparatus must never be put into the wicker draining-basket.*

16. **Platinum foil and Platinum wire** are cleansed by boiling them in hydrochloric acid, and then rinsing off the acid with water. The wire should then be strongly heated for some time in the blowpipe-flame, until, when it is wetted with pure strong hydrochloric acid and held in the Bunsen-flame, it no longer persistently colours the flame. If the tip of the wire cannot be cleansed in this way, it should be cut off. Commercial platinum sometimes persistently colours the flame: wire made from such platinum is useless for flame-coloration tests and for spectrum analysis.

Platinum-foil and wire should be kept in a small beaker containing strong hydrochloric acid, which has been diluted with sufficient water to prevent it from fuming. When the platinum is removed from the acid and has been rinsed with water, it will usually be found to be clean and to give no colour to the flame.

HEATING GLASS AND PORCELAIN.

17. **A few general Precautions** should be observed in heating glass and porcelain vessels, in order to guard against cracking them. The two following rules apply to both glass and porcelain :—

A vessel containing a liquid must never be heated by the flame above the level of the liquid inside.

A hot dry vessel must be allowed to cool before any liquid is poured into it, and before it is placed upon a cold surface.

18. **Porcelain Vessels** withstand higher temperatures than glass vessels, and are not so liable to be cracked by being suddenly heated or cooled.

Porcelain Dishes are generally used for the purpose of evaporating liquids to dryness, or for concentrating them. They are supported on a pipe-clay triangle, which is placed upon a tripod-stand or a retort-stand. Porcelain dishes may be safely heated by a small naked flame.

Porcelain Crucibles are used for strongly heating solid bodies. Crucibles are supported in the same way as porcelain dishes. The flame should not be allowed to play steadily upon the bottom of the crucible so as to heat it suddenly, but the burner should be constantly moved slightly from side to side until the porcelain is hot. The crucible should also be allowed to cool slowly on the triangle, since contact with a cold body is very apt to crack it. The hot crucible and cover are handled by the crucible-tongs.

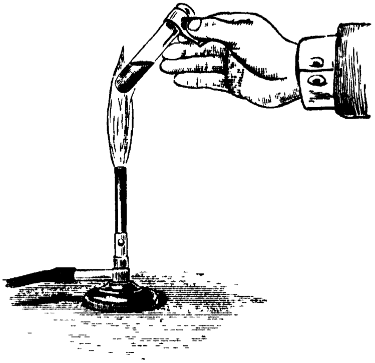
19. **Glass Vessels require to be Heated** more cautiously than porcelain vessels. A large naked flame must never be allowed to play for any length of time on one part of the glass surface. When a test-tube or boiling-tube is being heated, this *local heating* is prevented by holding the tube obliquely with the lower part in the flame, and either moving the tube

slowly up and down, or constantly turning it round on its axis (fig. 19).

Test-tubes and Boiling-tubes.—Ordinary test-tubes are too narrow to hold safely a large quantity of *boiling* liquid, since the upper portion of the liquid is liable to be projected from the tube during ebullition. A small quantity of liquid may be boiled, and a larger quantity may be heated short of boiling, in a test-tube. But the broader *boiling-tubes* are to be preferred when a liquid is to be boiled.

Test-tubes, or boiling-tubes, which are not full of liquid, can be held by the neck while they are being heated. They should be supported in an oblique position, so that the fingers are not over the flame (fig. 19). If steam or vapour is emitted in quantity, all risk of burning the fingers is avoided by bending round the neck of the tube a strip of folded *glazed* paper, or of leather, and pinching the ends of the strip together close to the tube, as is shown in the figure. This simple device serves better than any metal holder.

FIG. 19.



HOLDER FOR HEATING TUBE.

It is dangerous to use a strip of *unglazed* filter-paper, since if it becomes wetted it breaks and allows the tube to fall.

Crucible-tongs must never be used for holding test-tubes.

Glass Flasks, such as the wash-bottle, are most safely heated by placing them on a piece of wire-gauze on a tripod-stand over the Bunsen-flame (**1**), or they may be heated over the Fletcher flame (**2**, fig. 3). If a large sand-bath, hot plate, or water-bath (figs. 43, 45, **88**) is available, the flask may be heated on the hot surface or by the steam.

SECTION II.

PREPARATION AND PROPERTIES OF GASES—IDENTIFICATION OF GASES.

General Remarks.—In this Section full directions are given for the preparation of some of the more important gases, and for certain experiments which may be made with them. The experience thus gained enables the preparation of other gases to be carried out from a brief description. If a student has already prepared any of the gases the preparation need not be repeated.

Experiments which are described in small type may be omitted if time is limited, but the description should be carefully read through.

After the various gases have been prepared and the methods of detecting them have been tried, some mixtures of the gases should be examined with the view of detecting their constituents (73).

A full list of the apparatus and chemicals which are required for this Section will be found in the Seventh Section. The description of the fitting and of the use of the apparatus has been already given in the preceding paragraphs [1–19].

Directions for Working.—The student must carefully read through *the whole description* of each experiment before he begins to perform it. *Immediately after* the experiment has been successfully performed, a brief description of it should be entered in the Note-Book. Two students may work together through this Section; in the subsequent Sections, however, each student should work independently.

Caution.—Should any strong acid get upon the skin or the bench, it must be *at once* washed off. If it should be spilt upon the clothes, the part must be immediately wetted with ammonia solution : the red stain produced by acid on cloth is removed by ammonia unless it has been caused by nitric acid.

I.—PREPARATION AND PROPERTIES OF GASES.

OXYGEN GAS.

20. Preparation of Oxygen from Mercuric oxide.— When iron is exposed for some time to moist air, its surface becomes covered with rust. Many other metals undergo a similar change in moist air, but the alteration thus produced in their appearance is not usually as noticeable as in the case of iron. The liquid metal mercury does not rust at ordinary temperatures in air; but the metal becomes slowly covered with red mercury rust, when it is strongly heated for some time in a flask open to the air. This mercury rust has received the name of mercuric oxide. The fact that metals become heavier by rusting proves that something is added to them during the process. This is confirmed by the following experiment.

EXPERIMENT 1.—Place in a small perfectly dry test-tube sufficient mercuric oxide to cover the bottom. Heat the powder in the Bunsen-flame (I), covering the end of the tube loosely with the thumb, as is shown in fig. 20. As soon as small drops of mercury begin to appear upon the inside of the tube, remove the thumb, and quickly insert into the tube the burning end of a thin slip of wood. The flame will be seen to burn more brightly.

Heat the powder again for some time in the way just described, and then introduce the glowing end of the slip into the tube immediately after the flame has been blown out. The glowing end will burst into flame This behaviour with a glowing slip of wood is one of the most remarkable properties of oxygen gas, and is frequently used as a *test* for its presence. It is commonly known as the "spark test."

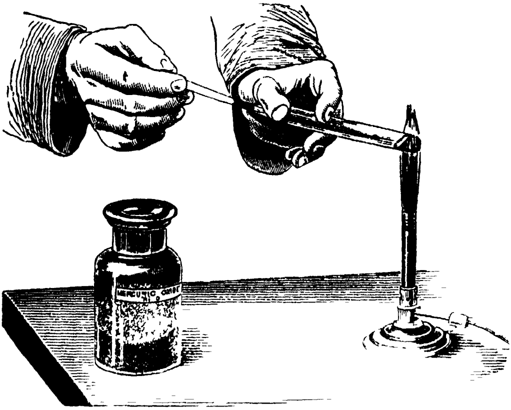
The small globules of mercury on the inside of the tube may be made to unite into larger ones by rubbing the surface with a glass rod.

Both the globules of mercury, and any red oxide which remains, should be preserved.

The chemical change which has occurred is thus represented by a chemical equation: $HgO = Hg + O$.

Since heat can separate mercuric oxide into mercury and oxygen, the rusting of mercury evidently consists in the metal taking oxygen gas from the air. This accounts for the increase of weight which the metal undergoes while it is rusting. If all metal-rusts could be decomposed by heat, oxygen might be obtained in the manner just described from any one of them.

FIG. 20.



PREPARATION OF OXYGEN.

The above method of making oxygen is instructive, and it is interesting because it was the first means known of preparing the gas. The method is, however, never used for the preparation of large quantities of oxygen, since other substances are known which contain a larger proportion of oxygen, and which give it off when they are heated more easily than mercuric oxide does. These substances are also preferred on account of their greater cheapness.

21. Preparation of Oxygen from Potassium chlorate.—Potassium chlorate is usually employed for the preparation of small quantities of oxygen. If this salt is heated until no more gas is given off, it parts with the whole of its oxygen, as is shown by the following equation: $KClO_3 = KCl + O_3$.

EXPERIMENT 2.—Heat a few crystals of potassium chlorate in a clean dry test-tube. The white salt *decrepitates* or crackles, and then *fuses* or melts. When the liquid salt is further heated it appears to boil. The small bubbles which are given off consist of oxygen gas. This may be readily proved by holding in the mouth of the test-tube a burning or glowing splinter of wood.

22. Preparation of Oxygen from Oxygen-Mixture.—Oxygen gas is much more readily expelled by heat from potassium chlorate than from mercuric oxide. But if the chlorate is mixed with a small quantity of some other suitable substance, its oxygen is driven off by heat with extreme facility. Manganese dioxide, or black oxide of manganese, is usually chosen for mixture with the chlorate. The manganese oxide does not itself seem to undergo any permanent change in the process.

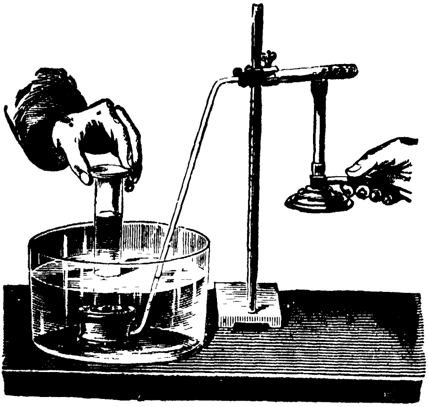
EXPERIMENT 3.—Powder finely in a mortar sufficient potassium chlorate to fill a watch-glass. Mix the powder in the mortar with about one-fifth as much powdered manganese dioxide. Heat a small quantity of this mixture in a test-tube. A comparatively gentle heat will cause the oxygen to be rapidly evolved, and the gas may be readily detected by the "spark-test" in the upper part of the test-tube.

23. Preparation and Collection of Oxygen.—In the preceding experiments the oxygen has been detected in the test-tube in which it was prepared, and has been allowed to pass away freely into the air. When the gas is to be collected in a vessel, the air is first removed from the vessel by filling it with water, and the gas is then allowed to displace the water.

In order to collect the gas, a bent-glass delivery-tube is fitted by means of a cork into the mouth of the test-tube (fig. 21). The end of this delivery-tube is immersed in water immediately beneath the mouth of the collecting-vessel, which has also been inverted with its neck in the water. When the test-tube is heated, the oxygen escapes from the end of the delivery-tube and passes as bubbles into the collecting vessel, gradually displacing the water from it.

This process of *collecting* oxygen is fully described in the following experiment.

FIG. 21.



PREPARATION AND COLLECTION OF OXYGEN.

EXPERIMENT 4.—Select a sound cork which is somewhat too large to fit the mouth of the test-tube to be used. Soften it by squeezing it, or by rolling it on the floor with gentle pressure from the foot. The cork should now fit tightly into the mouth of the test-tube.

Then bend (5) a piece of glass tubing, about fourteen inches in length, into the form shown in fig. 21. The bends should be so made, that when the apparatus is fitted together the test-tube is at a convenient height for being heated by the flame, while the end of the delivery-tube dips about an inch under the water contained in the glass or stoneware vessel.

Now make a hole through the centre of the cork (II) of such a size that the glass tube fits tightly into it. Then push the glass tube into the cork and the cork into the test-tube, and test whether the apparatus is air-tight by blowing down the open end of the delivery-tube. No air must be heard to escape, or must be seen to bubble out on moistening the cork. If air does escape, a fresh cork must be prepared.

Now transfer the oxygen-mixture (Exp. 3) into the perfectly dry test-tube, either by pouring it off a piece of paper folded into a trough, or by scooping it up from the mortar with the mouth of the test-tube. Then fit in the cork with the delivery-tube.

Before the test-tube is heated, fill the jar, in which the oxygen is to be collected, with water. Close its mouth with a ground-glass plate. Then invert its mouth in water, three or four inches in depth, contained in the stoneware or glass vessel, and carefully remove the glass plate. If this operation has been performed with proper care, the jar will be entirely filled with water, and no air-bubble will remain.

Next proceed to heat the front part only of the oxygen-mixture, gradually extending the heating towards the hinder portion as the evolution of gas slackens.

The test-tube may either be held in the hand or may be supported by a clamp in an almost horizontal position. The end containing the mixture should be somewhat raised, so as to prevent any condensed moisture from trickling back upon the heated portion and cracking it A small flame only should be used, in order to prevent any part of the glass from being too suddenly and strongly heated, which would be liable to crack or melt it. The flame should also at first be slowly moved from side to side for the same reason.

Oxygen gas will soon be evolved, but it will not at once appear at the end of the delivery-tube, since it has first to drive out the air which filled the apparatus. As soon as the glowing end of a slip of wood is kindled, when it is held at the mouth of the delivery-tube, the oxygen has driven out, the air and is beginning to escape. The end of the delivery-tube is then at once dipped under water beneath the mouth of the jar, and the stream of bubbles will rapidly rise in the jar and displace the water.

A stoneware beehive-shelf will be found convenient for supporting the jar in the bowl, as is shown in the figure, but the jar may instead be simply held in the hand.

As soon as the jar is full of gas, its mouth is closed under water with the glass plate, and it is removed for experiment. The jar may also be removed by slipping under its mouth a small dish or saucer; the water which is taken out in the saucer will then serve to close the mouth of the jar.

Three jars should be filled with oxygen for the following experiments. One vessel may, however, be refilled with the gas after the completion of each experiment, if a number of jars are not at hand.

If at any time the gas should be given off too rapidly, the flame must be removed until the current of gas slackens. The hinder portions of the mixture should be heated only after the part in front refuses to yield any more gas. When the process is to be stopped, the end of the delivery-tube must be removed from the water before the gas has ceased to bubble out. The hot test-tube must not be allowed to touch cold or wet objects, which would crack the glass.

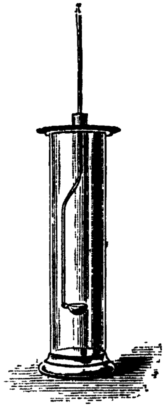
24. Combustions in Oxygen.—Oxygen gas is remarkable for the energy with which it combines with or burns many heated substances. Three examples of this property are given in the following experiments.

EXPERIMENT 5.—Select a piece of **wood-charcoal** about the size of a hazel-nut. Place this in a metal deflagrating-spoon. Then adjust the wire handle in the brass cap, so that when it is held beside the jar of oxygen with the cap on a level with its mouth, the spoon is about an inch from the bottom of the jar (fig. 22).

Now heat the charcoal in the Bunsen-flame or the blow-pipe-flame (6) until a part of its surface glows. Then quickly place it in the jar of oxygen with the brass plate of the spoon covering the mouth (fig. 22). The charcoal will burn much more brilliantly and rapidly than it does in air, and will convert the oxygen into carbon dioxide gas : $C + O_2 = CO_2$.

When the combustion ceases, pour into the jar some clear lime-water from a small beaker or test-tube. Then quickly close the jar and shake the liquid inside it. The clear lime-water will become milky. This indicates the presence of carbon dioxide gas, as will be hereafter explained.

FIG. 22.



COMBUSTION IN OXYGEN.

EXPERIMENT 6.—Remove the charcoal from the deflagrating spoon, and replace it by a piece of **sulphur** as large as a pea. Heat the spoon in the flame until the sulphur melts and begins to burn with a pale blue and almost invisible flame. Then place the spoon in a fresh jar of oxygen. The sulphur will at once burn with a much brighter flame, converting the oxygen into sulphur dioxide gas: $S + O_2 = SO_2$. The presence of this gas in the jar, after the combustion has ceased, is proved by its suffocating smell.

Pour a little water into the jar and shake it round ; sulphurous acid will at once be formed. This acid may be recognised by dropping into the water a piece of blue litmus-paper, which is immediately reddened ; also by pouring in *a few drops* of red potassium dichromate solution, the colour of which will be changed to green.

EXPERIMENT 7.—Kindle any sulphur, which may remain in the deflagrating spoon, by holding the spoon in the Bunsen-flame. As soon as the spoon is free from sulphur, and is again quite cold, place in it a small piece of carefully dried **phosphorus** not larger than half a pea.

The phosphorus may be cut with a knife. It must be touched only with the brass tongs or with *wet fingers*. Phosphorus should be handled as little as possible, since it is liable to be kindled by the heat of the hand, and it then causes dangerous wounds. This substance should always be kept under water, and should only be dried, immediately before it is used, by pressing it between filter-paper or blotting-paper or with a dry cloth.

Set fire to the phosphorus by holding the spoon in the flame, and notice that it burns brilliantly in the air. Then place the spoon in a jar of oxygen. The phosphorus will burn much more brilliantly. A white substance, called phosphoric oxide, is the product of combustion both in air and in oxygen: $P_2 + O_5 = P_2O_5$.

When the phosphorus has ceased to burn, pour a little water into the jar and shake it round. The white substance will dissolve in the water, yielding solution of phosphoric acid: $P_2O_5 + 3H_2O = 2H_3PO_4$. The water may be proved to contain the acid by dropping into it a piece of blue litmus-paper, which will be immediately reddened.

25. **Tests for Oxygen.**—Unless oxygen is largely diluted with other gases, it is easily detected by introducing into it a slip of wood with a spark at the end; the spark bursts into flame. This test only serves for the detection of oxygen in a mixture when its proportion in the mixture is large. Nitrogen monoxide gas also kindles a spark into flame, but this gas is readily distinguished from oxygen by a greenish halo appearing round the flame thus produced. (Exp. 50, p. 52.)

Another test for uncombined oxygen, when it occurs alone or in gaseous mixtures, depends upon its power of colouring nitric oxide gas reddish-brown (Exp. 27, p. 40). The test serves to distinguish oxygen from nitrous oxide gas, which gives no colour when it is mixed with nitric oxide.

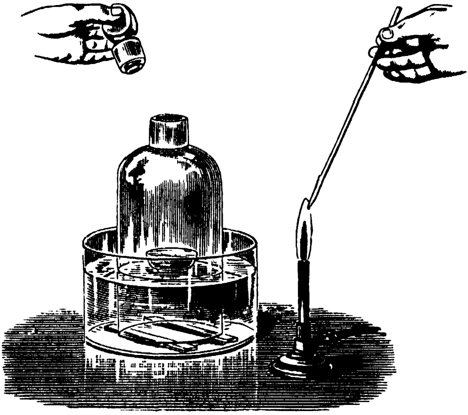
A freshly made mixture of solutions of potassium hydrate and pyrogallol absorbs oxygen and is coloured brown by it.

NOTE.—The above tests for oxygen are also yielded by ozone, but methods of distinction are given in par. 46.

NITROGEN GAS.

26. Preparation of Nitrogen from Air.—Atmospheric air consists mainly of a mixture of oxygen with nitrogen. In order to remove the oxygen and obtain the nitrogen, phosphorus may be burnt in some air confined over water. The phosphoric oxide thus formed is allowed to settle down and dissolve in the water. The volume of nitrogen which remains will be found to amount to four-fifths that of the air employed. The gas may be shown to have the property of extinguishing a flame.

FIG. 23.



PREPARATION OF NITROGEN FROM AIR.

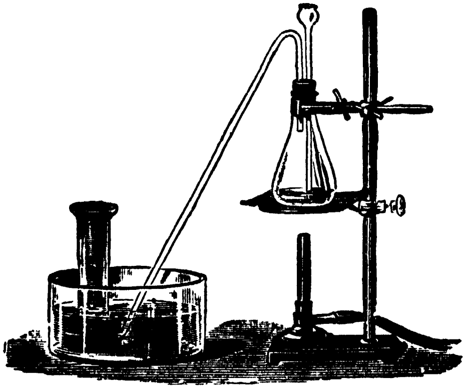
EXPERIMENT 8.—Place a small piece of well-dried phosphorus in a little dry porcelain dish and float the dish upon some water in a pan. Then place an open bell-jar as a cover over the floating dish, immersing the mouth of the bell-jar in the water, and allowing it to rest on two strips of sheet-lead which have been laid upon the bottom of the pan (fig. 23).

Kindle the phosphorus by touching it with the end of a long piece of wire, or of a glass rod which has been heated in the flame and then pushed down through the neck of the bell-jar. Immediately insert the cork or stopper, and hold down the jar until it stands steadily by itself.

When the phosphorus has united with all the oxygen of the inclosed air, it ceases to burn. Water will then gradually rise in the bell-jar, as the heated residual gas contracts by cooling. The water will ultimately rise in the bell-jar and fill one-fifth of the space which was previously occupied by the air.

Now pour water into the pan, until it stands at the same level both inside and outside the bell-jar. Remove the cork or stopper and introduce a burning taper or strip of wood. The flame will be at once extinguished by the nitrogen.

FIG. 24.



PREPARATION OF NITROGEN FROM AMMONIUM NITRITE.

27. Preparation and Properties of Nitrogen.—It has been recently shown that atmospheric nitrogen contains a small proportion of argon, and minute proportions of other gases. Pure nitrogen may be prepared by heating a solution of sodium nitrate and ammonium chloride. The chemical change is as follows :—

$$NaNO_2 + NH_4Cl = N_2 + NaCl + 2H_2O.$$

EXPERIMENT 9.—Fit a four-ounce flask with a doubly perforated cork (11). Pass a thistle-funnel through one hole in the cork, letting it reach nearly to the bottom of the flask. Through the other hole pass a delivery-tube bent (5) to the shape shown in fig. 24, and prove the apparatus to be sound by closing the end of the delivery-tube and blowing down the thistle-funnel Arrange this apparatus for collection of the gas as is shown in fig, 24.

Introduce about 5 grams of sodium nitrite and 5 grams of ammonium chloride into the flask, add 50 c.c. water, and gently heat the mixture. When the air has been expelled from the apparatus, invert over the beehive cell a glass cylinder, which has been previously filled with water and closed with a glass plate, and remove the plate.

The gas will gradually displace the water. As soon as the jar is full of gas, slide a glass plate over its mouth while it is immersed in the water and remove the jar. Fill two more jars, and use one jar for each of the following tests.

28. Tests for Nitrogen.—Notice that the gas is colourless, and possesses no smell; that it at once extinguishes a lighted taper; and that when lime-water is shaken in the jar, the liquid remains clear.

HYDROGEN GAS.

29. Preparation of Hydrogen from Hydrochloric acid.—Water consists of oxygen combined with another gas called hydrogen. Several processes are known for preparing hydrogen from water. Hydrogen is, however, also a constituent of all acids, and it is most readily obtained from certain acids by the action of metals upon them.

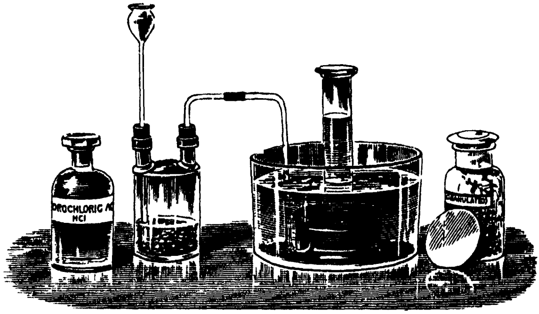
EXPERIMENT 10.—Fit the necks of a two-necked Woulffe's bottle with perforated corks, one of which bears a thistle-funnel whose end reaches nearly to the bottom of the bottle, and the other a bent tube terminating just below the cork (fig. 25). Then attach a bent delivery-tube to this tube by means of a short piece of tightly-fitting rubber tubing.

A wide-necked bottle, fitted as is shown in fig. 29 (p. 34), may be used instead of the Woulffe's bottle.

Put into the bottle sufficient granulated zinc to cover the bottom, and replace the cork. Then ascertain that the apparatus is air-tight by closing the end of the delivery-tube and blowing down the thistle-funnel. Now pour water in through the funnel, until the zinc and the end of the funnel-tube are covered; then add pure strong hydrochloric acid gradually (see Caution, p. 18). Hydrogen will be seen rising from the zinc in numerous small bubbles: $Zn + 2HCl = H_2 + ZnCl_2$.

30. Collection of Hydrogen.—Dip the end of the delivery-tube under water contained in a pan, and allow the gas to bubble out through the water for at least five minutes. This delay in collecting the gas is necessary in order to give the hydrogen time to remove the air which filled the bottle.

FIG. 25



PREPARATION AND COLLECTION OF HYDROGEN.

It should be remembered that a mixture of air with hydrogen burns explosively when it is kindled. The freedom of the escaping gas from air should therefore be ascertained as follows. Invert a test-tube filled with water over the end of the delivery-tube. When the test-tube is full of gas close its mouth with the thumb, and remove the tube from the water. Then hold its open mouth to a flame. If the gas burns with a slight explosion, the tube must be filled again with the gas and the experiment repeated.

As soon as the gas burns quietly within the test-tube, a small glass cylinder may be filled with water, then inverted in the pan of water and filled with hydrogen (fig. 25).

During the further preparation of gas for the following experiments, it will be found occasionally necessary to pour in a little strong hydrochloric acid through the funnel-tube, in order to render the evolution of hydrogen more rapid.

31. Hydrogen is lighter than Air.—It is easy to show that hydrogen rises in the air in the following way.

EXPERIMENT 11.—Hold a jar of hydrogen for a short time with its mouth open and directed upwards. Then introduce a lighted taper into the vessel. No hydrogen flame will be seen, and no explosion will occur. This proves that the hydrogen has entirely escaped from the jar.

Then fill the cylinder once more with hydrogen, and hold it for a short time with its mouth downwards. Now push up a lighted taper into the jar. The hydrogen will burn with a pale flame. Note, however, that the gas burns in this case with a slight report; this is due to air having mingled to some extent with the hydrogen by spontaneous *diffusion.*

Since hydrogen has not escaped downwards, while it has rapidly escaped upwards, it is proved to be lighter than air.

32. Collection of Hydrogen by Displacement.—Advantage is often taken of the lightness of hydrogen to facilitate the process of its collection. The gas is allowed to directly replace the air from the vessel in which it is to be collected. For this purpose a straight delivery-tube from the hydrogen-apparatus is passed up to the top of the inverted vessel. The hydrogen then collects in the upper part of the vessel, and gradually pushes out the heavier air.

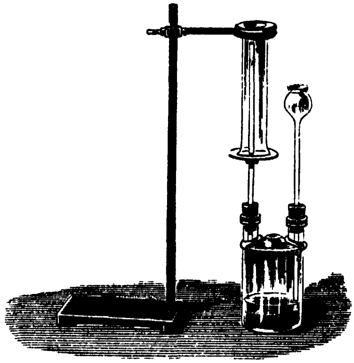
EXPERIMENT 12.—In order to fill a jar with hydrogen by this process of *direct displacement*, quickly replace the bent tube in the cork of the hydrogen generator by a straight tube eight or nine inches in length (fig. 26). Then cause a brisk effervescence of gas to take place by pouring some strong hydrochloric acid down the funnel-tube, and pass the delivery-tube up to the top of an inverted jar. Allow the jar to remain in this position for several minutes.

The mouth of the jar may be loosely closed by a piece of perforated metal or cardboard, which rests upon a small piece of india-rubber tubing pushed on the delivery-tube. This will hinder the spontaneous mixture with the air, or *diffusion*, which occurs when the hydrogen is in contact with the air.

33. Hydrogen is Combustible but Extinguishes Flame.—Prove this by means of the jar which has just been filled by displacement.

EXPERIMENT 13.—Remove the jar (fig. 26), keeping it inverted, and push up inside it a burning wax taper, five or six inches in length. The hydrogen will be kindled, and will burn with a pale flame at the mouth of the jar, but the flame of the taper will be seen to be extinguished by the gas. The taper may, however, be rekindled by holding its end in the hydrogen flame which is burning at the mouth of the jar.

FIG. 26.



COLLECTION BY DISPLACEMENT

34. Hydrogen forms Water when it is Burnt in Air.—This is proved by burning the hydrogen as it escapes from the apparatus, and then condensing the steam which is formed by the combustion as it escapes from the flame.

EXPERIMENT 14.—Cover the generating bottle and funnel-tube with a cloth in order to prevent danger in case of an explosion, and light the hydrogen at the end of the delivery-tube (fig. 26). Then hold over the flame a tumbler or beaker, which is perfectly clean, dry, and cool. The inside of the glass will become dimmed with moisture. This shows that when hydrogen burns in the air, it is uniting with oxygen and producing water: $H_2 + O = H_2O$.

In order to make the above result conclusive the hydrogen should be dried before it is burnt, since it will always contain more or less vapour of water derived from the liquid in the generator. The gas may be dried or *desiccated* by passing it through some substance which readily absorbs moisture, such as strong sulphuric acid or fragments of solid calcium chloride. A straight tube containing fragments of calcium chloride (fig. 27 *a*), or a U-tube filled with pieces of pumice-stone moistened with strong sulphuric acid (fig. 27 *b*), may be connected with the hydrogen-apparatus for the purpose of drying the gas. A more convenient form of U-tube is shown in fig. 28.

FIG. 27.

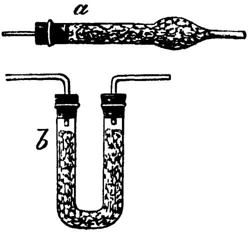
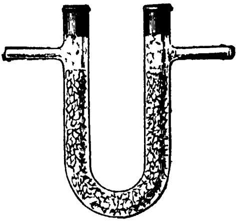


FIG. 28.



DIFFERENT FORMS OF DRYING-TUBES.

35. **Explosion of a Mixture of Hydrogen and Oxygen.**—A mixture of hydrogen with half its volume of oxygen burns with violent explosion when it is kindled.

EXPERIMENT 15.—Fill a short thick-glass cylinder with water, then invert it in a pan of water. Displace one-third of the water in the cylinder with oxygen (23, Exp. 4), and the remainder with hydrogen (29, Exp. 10). Let the jar stand with its mouth under water for five or six minutes in order to allow the gases to mix. Then raise the inverted jar and apply a lighted taper to its mouth The mixture of gases will burn rapidly with a loud explosion.

36. **Tests for Hydrogen.**—Hydrogen gas is recognised by burning with a pale flame when it is kindled in air or in oxygen. The flame of the dried gas deposits water on the inside of a clean cold glass vessel. No carbon dioxide is formed during the combustion of the gas (39).

When hydrogen is mixed with half its volume of oxygen, or with a corresponding volume of air, and the mixture is kindled, it burns explosively with formation of moisture.

CARBON DIOXIDE, OR CARBONIC ANHYDRIDE GAS.

37. Preparation of Carbon Dioxide from Marble.

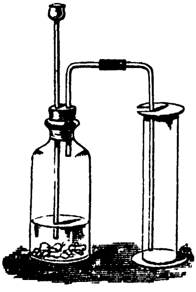
When carbon is burnt in oxygen (24, Exp. 5) carbon dioxide gas remains in the jar, mixed with nitrogen. This gas is easily obtained, unmixed from nitrogen or other gases, by pouring hydrochloric acid upon some pieces of marble:

$$CaCO_3 + 2HCl = CO_2 + H_2O + CaCl_2.$$

Chalk or limestone may be substituted for marble, but it will usually froth and give off gas too rapidly.

EXPERIMENT 16.—Rinse out the hydrogen apparatus (fig. 25, p. 30), or fit up a special bottle (fig. 29), and place in it some small pieces of marble. Push into the rubber joint a delivery-tube bent at right angles (5), as is shown in fig. 29. Then pour through the funnel-tube sufficient water to cover the marble and the end of the funnel-tube, and add strong *commercial* hydrochloric acid until the gas comes off with brisk effervescence. Place the delivery-tube in a dry jar, with its end nearly touching the bottom, and allow the gas to pass into the jar for several minutes.

FIG 29.



PREPARATION OF CARBON DIOXIDE.

Carbon dioxide is much heavier than air, and it will therefore soon fill the jar by displacement; that is to say, the gas which passes constantly to the lower part of the jar will remain there, and will gradually lift out the air.

This gas has the property of extinguishing a flame, and it is, therefore, easy to ascertain when the jar is full, by holding a lighted taper just inside its mouth: if the flame is extinguished, the carbon dioxide is shown to have reached the top of the jar.

It is well to prevent diffusion by covering the mouth of the jar with a small disc of cardboard, which has had a slit or hole cut in it for the delivery-tube.

Carbon dioxide is largely dissolved by water, hence the process of collecting it by direct displacement is preferred to collection over water, which would entail some loss of gas.

38. Carbon dioxide is Heavier than Air. Prove that it tends to fall in the air in the following manner.

EXPERIMENT 17.—Allow a jar of carbon dioxide to stand uncovered with its mouth upwards for a few minutes. Then introduce a burning taper into the jar: the flame will be immediately extinguished. This proves that the carbon dioxide is still present in the jar.

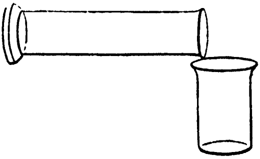
Now hold the jar for several minutes with its mouth downwards, and test with a lighted taper; the flame of the taper will continue to burn inside the jar, showing that the carbon dioxide has been replaced by air.

These experiments prove that carbon dioxide is heavier than air, since it has remained in the vessel which is open above and has fallen out of the one which is open below.

EXPERIMENT 18.—As this gas is much heavier than air, it can be poured like water from one vessel into a second vessel which is full of air.

A jar full of carbon dioxide is gradually tilted a little beyond the horizontal position, with its mouth over that of a small beaker (fig. 30). After the jar has been held in this position for a short time, it may be shown by means of a lighted taper that the gas has left the jar and is present in the beaker.

FIG. 30.



POURING OUT CARBON DIOXIDE.

39. Carbon dioxide turns Lime-water milky.—This is the usual test for detecting the presence of the gas.

EXPERIMENT 19.—Pour some lime-water from a test-tube or small beaker into a jar of carbon dioxide, and shake the liquid round in the jar. The lime-water will at once become milky.

The lime, which was dissolved in the water and was therefore invisible, has been converted by the carbon dioxide into chalk or calcium carbonate; and this, being an insoluble substance, remains mixed as a white powder or *precipitate* with the water: $CaH_2O_2 + CO_2 = CaCO_3 + H_2O$.

40. Carbon dioxide dissolves in Cold Water, and is removed by Boiling the Water. The solution of the gas probably contains carbonic acid: $CO_2 + H_2O = H_2CO_3$.

The solubility of the gas in water, and its removal from solution by boiling the water, may be proved as follows.

EXPERIMENT 20.—Carbon dioxide is made to displace the air from a jar which has been previously half filled with cold water (fig. 29). The mouth of the jar is then tightly closed with the wetted palm of the hand, and the gas and water are shaken together vigorously for a short time. The mouth of the jar will now firmly adhere to the hand, owing to the reduction of the pressure of the gas caused by its absorption by the water.

EXPERIMENT 21.—A further proof of the solubility of carbon dioxide is afforded by dipping the end of the delivery-tube (fig. 29) to the bottom of a small beaker containing water, so as to cause the gas to bubble through the liquid. After the bubbles have passed through the water for several minutes, a few drops of the liquid may be poured into a test-tube containing lime-water; the lime-water will immediately become milky.

If a little blue litmus solution, or a piece of blue litmus-paper, is dropped into another part of the carbonic acid solution, the litmus will become red, indicating the presence of an acid. Preserve this portion of the liquid for Exp. 22.

The liquid which contains carbonic acid will be found to possess a taste resembling that of soda-water. In fact, soda-water is merely water which has been made to dissolve a large quantity of carbon dioxide, by shaking up the water with the gas under pressure. By testing soda-water with lime-water and with litmus, it may be readily shown to contain carbonic acid.

Experiment 22.—Heat some of the water which contains carbonic acid (Exp. 21) in a test-tube until it boils. As soon as the heat is applied, bubbles of gas will rise in the water; and after it has been boiled briskly for several minutes, the liquid may be proved to be free from carbonic acid by testing it with lime-water and with litmus.

The reddened litmus in the carbonic acid solution, which was put by from a previous test (Exp. 21), will also become blue when the solution is boiled.

These experiments prove that carbon dioxide may be completely expelled from water by boiling the liquid for some time.

41. Calcium carbonate dissolves in Carbonic acid solution, and is Precipitated by Boiling the liquid.

Experiment 23.—Dilute some lime-water in a small beaker with about an equal measure of distilled water, and allow carbon dioxide gas to bubble through the liquid. Milkiness will be produced at first, owing to the formation of calcium carbonate. But if the gas is allowed to pass for several minutes through the liquid the milkiness will gradually disappear, showing that calcium carbonate is soluble in carbonic acid.

When this clear liquid, however, is boiled for a short time, it will become milky again, because the chalk can no longer remain dissolved after the carbonic acid has been removed (Exp. 22).

When the water is poured out, part of the calcium carbonate will be seen adhering to the inside of the tube; it may be readily removed by rinsing out the tube with a little hydrochloric acid.

The above experiment explains the origin of the coating or *incrustation* which may be seen inside kettles and steamboilers, in which hard chalk-water has been boiled for some time. Such water contains chalk dissolved by carbonic acid; this acid is removed when the water is boiled, and a great part of the chalk is deposited upon the inside of the vessel.

42. **Tests for Carbon dioxide.**—The presence of carbon dioxide gas may be detected by its power of extinguishing a burning taper, and of turning lime-water milky. The latter test distinguishes it from nitrogen and from certain other gases which extinguish flame.

Detection of Carbon dioxide in the Breath, from a Flame, and in the Air.—The above tests may be employed to prove that carbon dioxide is evolved from the lungs during the process of respiration, and also that it is produced by an ordinary flame. The gas is, therefore, being constantly introduced into the air, and it may always be detected in the air by the lime-water test.

EXPERIMENT 24.—Invert a jar filled with water in a pan of water, and blow the breath up into the jar through a glass tube, one end of which is dipped beneath the mouth of the jar.

In order to obtain air *from the lungs*, take a full breath; then expel the greater part of the breath, so as to replace the air contained in the windpipe and mouth by air from the lungs. Allow the remainder of the breath to bubble up into the jar.

Now close the jar, remove it from the pan, and introduce into it a lighted taper: the flame will be immediately extinguished.

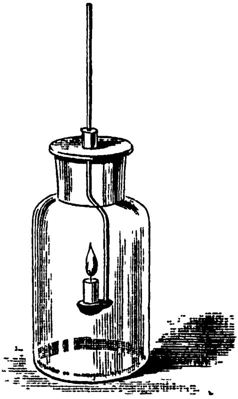
Fill another jar in the same way and shake up lime-water in it. Also blow the last part of the breath through a glass tube into lime-water contained in a small beaker. The lime-water will become milky in each case.

EXPERIMENT 25.—Light a small piece of candle or wax-taper upon a deflagrating-spoon, and place it in a bottle of air, the mouth of which is closed by the brass plate of the spoon (fig. 31).

The flame will burn for a short time and will then be extinguished. If the candle is relighted and replaced in the bottle, the flame will be immediately extinguished. The extinction of the flame is due to the presence of carbon dioxide, and to the reduction of the amount of free oxygen in the air, which result from the combustion of the candle.

Now pour some lime-water from a small beaker into the bottle and shake the liquid round. It will become milky.

FIG. 31.



CANDLE IN CLOSED BOTTLE.

A similar experiment may be performed by inverting the bottle over a small gas-flame, closing the mouth of the bottle with a glass plate as soon as the flame is extinguished, and then shaking lime-water in the bottle. The lime-water will become milky, proving that carbon dioxide is produced by the combustion of coal-gas.

EXPERIMENT 26.—Pour some clear lime-water into a watch-glass or clock-glass, and allow it to stand for a few minutes exposed to the air. A film of calcium carbonate will gradually form on the surface of this liquid and render it dull: the film will be broken up into white flakes when the water is stirred. This proves the presence of carbon dioxide in the air.

NITRIC OXIDE GAS.

43. Preparation of Nitric oxide from Nitric acid.—This gas may be prepared by the action of diluted nitric acid on copper: $3Cu + 8HNO_3 = 2NO + 3Cu(NO_3)_2 + 4H_2O$.

EXPERIMENT 27.—Cleanse the apparatus which has been already used for the preparation of hydrogen (29, Exp. 10), place some copper clippings or turnings in it, and pour in water until the end of the funnel-tube is covered. Then add strong commercial nitric acid gradually (see Caution, p. 18), until a reddish-brown gas appears inside the bottle. Allow this gas to bubble off through water. After a short time the colour will almost entirely disappear, and a colourless gas may then be collected over water in the same way as hydrogen gas was collected (30).

44. **Tests for Nitric oxide.**—Nitric oxide is colourless; but when it is mixed with free oxygen, nitric oxide is converted into reddish-brown nitrogen peroxide

EXPERIMENT 28.—Invert a jar filled with water in water, and fill it with nitric oxide gas. Then either pass oxygen up into it, or remove the jar from the water and allow it to stand with its mouth open to the air The entrance of the oxygen, or of the air, into the nitric oxide will at once produce a reddish-brown colour.

The origin of the coloured gas which was seen in the preparation-bottle in Exp. 27 is now explained. The bottle was at first filled with air; as soon as the first portions of the nitric oxide gas were produced, they therefore mingled with the free oxygen of the air and formed nitrogen peroxide.

This coloured gas is produced as long as any free oxygen remains in the bottle. The coloured gas is rapidly dissolved by water.

OZONE GAS.

45. **Preparation of Ozone from Air.**—Oxygen is converted by the influence of electricity into a gas known as ozone. When certain processes of oxidation are being effected by free oxygen, the oxygen is also partially converted into ozone. Ozone has a peculiar smell, and possesses other properties which distinguish it from oxygen. Since, however, ozone can be made from oxygen and can be reconverted into oxygen without any change of weight occurring, ozone is only altered or *allotropic* oxygen. The preparation of ozone by the spontaneous oxidation of phosphorus in the air is described below.

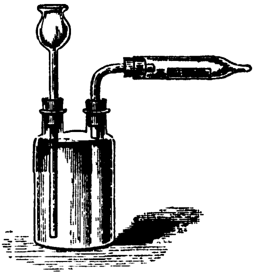
The student may save time by reading through Exp. 29 and trying the test for ozone by placing phosphorus in a flask (Exps. 30, 31).

EXPERIMENT 29.—Cleanse the apparatus which was used for preparing nitric oxide (44, Exp. 27), and replace the rubber joint and the bent delivery-tube by a test-tube, the bottom of which has been drawn out into an open nozzle (fig. 32). The joint may be made by a piece of rubber tube or by a perforated cork.

Now place in this tube a stick of phosphorus, about an inch in length, and pour water slowly in through the funnel-tube so as to drive the air from the interior of the bottle over the phosphorus and out through the nozzle.

The escaping air will be found to have the peculiar smell of ozone, showing that the oxygen of the air has been partly converted into ozone by contact with the phosphorus.

FIG. 32.



PREPARATION OF OZONE.

46. **Tests for Ozone**.—Ozone answers to the tests for oxygen (25), but is distinguished by its smell and by separating iodine from potassium iodide. The presence of the iodine thus liberated is confirmed by its power of turning starch solution blue.

EXPERIMENT 30.—Stir a little starch-powder with some cold water and pour this gradually into some water which is boiling briskly in a small porcelain dish · then cool the liquid. Mix a few drops of potassium iodide solution with part of this starch-solution in the porcelain dish, and moisten a strip of filter-paper with the liquid. The paper thus prepared will serve to detect ozone, since the iodine which is separated by this gas from the potassium iodide, will strike an intense blue colour with the starch. Prove this by letting the stream of ozonised air, prepared as is directed above (Exp. 29) impinge upon this test-paper. If the air fails to turn the paper blue, repeat the experiment according to the directions given below (Exp. 31).

EXPERIMENT 31.—Occasionally it will be found difficult or impossible to obtain the ozone reaction with the apparatus described in Exp. 29. The method of experiment should then be modified by placing a few pieces of phosphorus at the bottom of a small glass flask, and hanging a strip of the moist ozone test-paper (Exp. 30) inside the flask. The neck of the flask is then closed by a watch-glass, and the test-paper is observed at intervals. In a period, which may vary from about five to twenty minutes. the change of colour indicative of the production of ozone will take place in the test-paper.

HYDROGEN SULPHIDE GAS.

47. **Preparation of Hydrogen sulphide.**—This gas is often called sulphuretted hydrogen.

EXPERIMENT 32.—It may be prepared in the apparatus which was used for the preparation of carbon dioxide (37, Exp. 16), and may be collected by downward displacement. Small pieces of ferrous sulphide are introduced into the cleansed apparatus, and diluted hydrochloric acid is poured upon them through the funnel-tube . $FeS + 2HCl = H_2S + FeCl_2$.

The gas is very poisonous and has a foul smell, it must therefore be prepared in the draught-closet. A mixture of this gas with air burns explosively when it is kindled.

The gas should if possible be collected from the general laboratory apparatus,.which is placed in a suitable draught-closet

48. **Tests for Hydrogen sulphide.**—Hydrogen sulphide is recognised by its peculiar and offensive smell, by its power of converting a colourless salt of lead into brown lead sulphide, and by forming pungent sulphur dioxide when it is burnt.

EXPERIMENT 33 —Moisten a piece of filter-paper with lead acetate solution, and expose the paper to the gas · it will become dark brown.

Burn the gas in a glass cylinder and at once close the cylinder with a glass plate. Note the deposition of yellow sulphur on the inside of the glass; this is due to the supply of oxygen being insufficient to secure the complete combustion of the gas. Slip aside the glass plate for a moment, and pour in quickly some water, which has been faintly coloured with potassium dichromate solution. Shake this liquid about in the jar. The colour of the liquid will change to green, showing that sulphur dioxide is present. This gas is thus proved to be a product of the combustion of hydrogen sulphide.

AMMONIA GAS.

49. Preparation of Ammonia from Ammonium chloride.

—The pungency of common *Smelling-Salts* is due to the ammonia gas which is constantly escaping from solid *Carbonate of ammonia.*

The gas is evolved much more rapidly if either *Carbonate of ammonia* or *Sal ammoniac* is mixed with slaked-lime, and the mixture is then gently heated.

EXPERIMENT 34.—Powder a little ammonium chloride or *Sal ammoniac* in a mortar, and mix with it about an equal quantity of finely powdered slaked-lime. Ammonia gas will be evolved from this mixture, and may be readily detected by its pungent smell:

$$2HN_4Cl + CaH_2O_2 = 2NH_3 + CaCl_2 + 2H_2O.$$

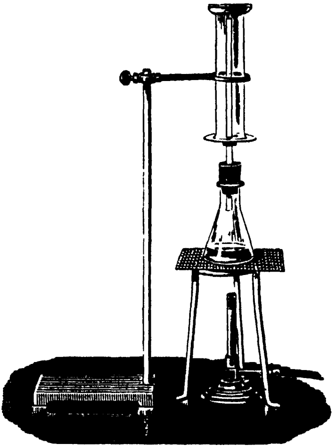
50. Preparation of Ammonia from Solution of Ammonia.—Ammonia gas is readily expelled from its solution in water by means of heat. This forms a convenient method of obtaining the gas.

EXPERIMENT 35.—Pour some strong solution of ammonia (*Liquor Ammoniae Fortissima*) into a small flask (fig. 33), until the flask is about one-quarter filled. Close the neck of the flask with a tightly-fitting perforated cork, into which a straight piece of glass tube, eight or nine inches long, has been fitted. Then place the flask upon a piece of wire-gauze on a tripod-stand, and heat it gently by a small flame.

Ammonia gas will at once escape from the liquid with effervescence, and will issue from the end of the tube.

Since ammonia is much lighter than air and is very soluble in water, it should be collected in a *perfectly dry* jar by the direct displacement of the air. The jar is conveniently supported over the delivery-tube by passing it through the ring of a retort-stand, and allowing its mouth to rest upon a cardboard disc, which is supported on a ring of india-rubber tubing slipped upon the delivery-tube (fig. 33).

FIG. 33.



PREPARATION AND COLLECTION OF AMMONIA.

In order to ascertain when the vessel is filled with the gas, it is only necessary to hold at the mouth of the jar a piece of red-litmus or yellow turmeric-paper. If the ammonia has filled the jar, it will change the colour of the litmus to blue and that of the turmeric to reddish-brown.

51. Combustion of Ammonia.—Ammonia gas does not burn continuously in cold air, but it burns readily in strongly heated air or in oxygen gas.

EXPERIMENT 36.—Bring a Bunsen gas-flame close to the end of the delivery-tube (fig. 33), from which a stream of ammonia is issuing. A pale yellowish-green flame of burning ammonia will be seen at the end of the tube as long as the air around is heated by the flame.

If the ammonia is kindled, as the end of the delivery-tube is pushed up into a jar of oxygen, the ammonia will also continue to burn in the oxygen.

52. Solubility of Ammonia in Water.—Ammonia is extremely soluble in water, and must therefore be collected either by displacement or over mercury. For this reason the inside of a vessel in which ammonia is to be collected should always be perfectly dried, in order to prevent loss of the gas from occurring by its absorption by the moisture. The solubility of ammonia in water may be proved by the following experiments.

EXPERIMENT 37.—Place a jar filled with ammonia mouth downwards in a vessel of water, and gently shake the jar so as to agitate the water at its mouth. The liquid will rapidly absorb the gas, and will rise in the jar until it fills the space which was occupied by the ammonia. A small part of this space, however, frequently remains filled with air which had become mixed with the ammonia during the process of charging the jar.

EXPERIMENT 38.—Pour a little water rapidly from a small beaker into a jar of ammonia gas, and at once cover the jar with a glass plate or with the hand. Then shake the water round in the jar and pour it out. The liquid will be found to have acquired the pungent smell, and the action upon litmus-paper and turmeric-paper, which characterise ammonia gas.

This liqnid is in fact weak *Liquor Ammoniae*, a solution which is prepared commercially in large quantities by passing ammonia gas for some time into cold water. Even at ordinary temperatures this solution slowly gives off a portion of the gas which it contains, and it furnishes ammonia gas readily when it is heated (Exp. 35).

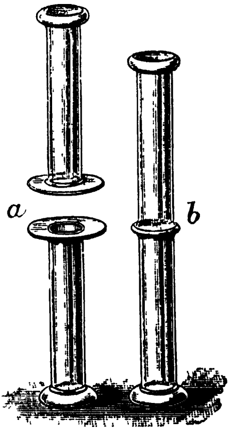
53. Ammonia gives White Fumes with Hydrogen chloride gas.—These fumes consist of particles of solid ammonium chloride.

EXPERIMENT 39.—Pour a little strong hydrochloric acid into a glass jar (see Caution, p. 18); close the mouth of the jar with a glass plate, and shake up the acid. Hydrogen chloride gas will thus be liberated, and will mingle with the air contained in the jar. Allow the liquid to run out of the jar by slipping aside the glass plate for a moment.

Place this jar in an inverted position over another jar, which contains ammonia gas and is also covered with a glass plate (fig. 34 *a*).

Then withdraw the glass plates, so that the mouths of the jars are in contact (*b*) and the hydrogen chloride and ammonia gases can freely intermingle. Dense, opaque, white fumes of solid ammonium chloride will immediately be formed: $NH_3 + HCl = NH_4Cl$.

FIG. 34.



AMMONIA AND HYDROGEN CHLORIDE.

These fumes may also be obtained by dipping a glass rod or a strip of filter-paper into some strong hydrochloric acid, and then holding it in the ammonia gas as it issues from the delivery-tube of the apparatus (fig. 33). The wetted rod or paper may also be pushed up into an inverted jar previously filled with the gas, or may be held at the mouth of a bottle containing strong solution of ammonia.

54. Tests for Ammonia gas.—Ammonia gas may be readily recognised by its pungent smell; by turning moistened red litmus-paper blue; by turning moistened turmeric-paper reddish-brown; and by giving opaque white fumes when it is brought into contact with a surface which has been moistened with *strong* hydrochloric acid.

CARBON MONOXIDE GAS.

This gas is produced when carbon dioxide is passed over red-hot charcoal: $CO_2 + C = 2CO$. It is often formed in this way in open grates, and is therefore seen burning with its characteristic blue flame at the upper surface of the glowing fuel. The gas is poisonous when it is breathed in any quantity.

Two methods for preparing carbon monoxide are described (55, 56). The gas is most easily made by heating sodium formate with sulphuric acid (56); but the preparation from oxalic acid (55) is also described, because it furnishes a good illustration of the ordinary method of purifying a gas by washing it with a suitable liquid, or by passing it over fragments of a suitable solid.

55. **Preparation of Carbon monoxide from Oxalic acid.**—A mixture of carbon monoxide with an equal measure of carbon dioxide may be prepared by heating crystals of oxalic acid with strong sulphuric acid:

$$H_2C_2O_4.2H_2O + H_2SO_4 = CO + CO_2 + H_2SO_4.3H_2O$$

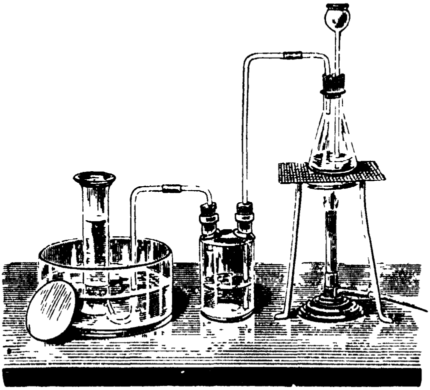
EXPERIMENT 40.—Pour upon a few crystals of oxalic acid, contained in a boiling-tube, about twice their volume of strong sulphuric acid, and heat the mixture (see Caution, p. 18). In a short time effervescence will commence, showing that gas is being evolved.

Introduce into the mouth of the boiling-tube a glass rod, which has been freshly dipped into lime-water; the drop of lime-water hanging upon its end will become milky, proving that carbon dioxide is present in the escaping gas. Hold the mouth of the boiling-tube against a Bunsen-flame, a blue flame will appear at the mouth of the tube, showing that carbon monoxide gas is also being evolved.

In order to remove the carbon dioxide and to obtain pure carbon monoxide, the mixture of gases is either *washed* with solution of caustic soda, or is passed over pieces of solid caustic soda or soda-lime. Caustic soda absorbs the carbon dioxide readily, but it does not affect carbon monoxide and allows it to pass on:

$$CO + CO_2 + 2NaHO = CO + Na_2CO_3 + H_2O.$$

FIG. 35.



PREPARATION AND COLLECTION OF CARBON MONOXIDE.

EXPERIMENT 41.—Heat a mixture of oxalic acid crystals and strong sulphuric acid in a flask fitted as is shown in fig. 35. Remove the flame as soon as effervescence commences, else the acid will froth over; then heat gently.

Allow the gases which are evolved to pass through a wash-bottle containing caustic soda solution, or through a suitable tube filled with fragments of soda-lime or with fragments of pumice-stone moistened with strong caustic soda solution. The bulbed tube (*a*) or the U-tube (*b*), shown in figs. 27, 28 (p. 33), are commonly employed as receptacles for the solid reagents. If the carbon dioxide is to be completely removed, the gases must be passed through two or more such tubes or bottles, and the stream of gas must be slow.

The carbon monoxide gas may be collected over water. It will produce no milkiness when it is shaken with lime-water, unless the stream of gas has been so rapid that the caustic soda has not been able to absorb the carbon dioxide completely.

56. **Preparation of Carbon monoxide from Sodium formate.**—When sodium formate is heated with strong sulphuric acid, carbon monoxide alone is given off:

$$NaHCO_2 + H_2SO_4 = CO + NaHSO_4.H_2O.$$

Carbon monoxide is thus readily obtained free from carbon dioxide. The preparation may be carried out in the apparatus shown in fig. 35, but the washing bottle may be dispensed with.

57. **Carbon monoxide Burns in Air, forming Carbon dioxide.**—Carbon monoxide resembles hydrogen by being inflammable and by extinguishing flame. It also burns explosively, when it is mixed with a suitable proportion of oxygen or of air and is then kindled. Hence, before this gas is collected in any quantity, it should be proved to be free from air by filling a small test-tube and seeing that the gas burns quietly when it is kindled.

EXPERIMENT 42.—Push a burning taper up into an inverted jar filled with carbon monoxide. The gas will burn with a blue flame at the mouth of the jar, but the flame of the taper will be extinguished.

As soon as the gas has ceased to burn inside the cylinder pour in a little lime-water and shake it about. The liquid will become milky, showing that the combustion of carbon monoxide in the oxygen of the air has produced carbon dioxide gas: $CO + O = CO_2$.

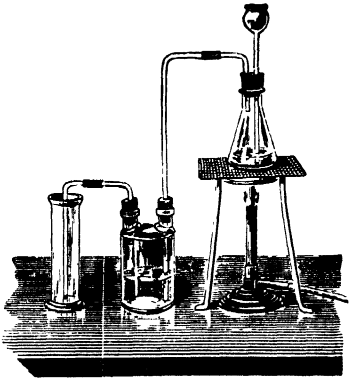
58. **Test for Carbon monoxide.**—Carbon monoxide is

recognised by burning with a pale blue flame in the air, producing carbon dioxide, which renders lime-water milky.

CHLORINE GAS.

59. **Preparation of Chlorine from Hydrochloric acid.**—This gas may be made in the apparatus which has been employed for the preparation of carbon monoxide (fig. 35, p. 47). The washing-bottle may either be dispensed with, or it may be partly filled with water in order to free the chlorine from hydrogen chloride gas : and the delivery-tube should not be curved round at the end, as it is not to be passed under water (fig. 36).

FIG. 36



PREPARATION AND COLLECTION OF CHLORINE.

EXPERIMENT 43.— Place some small pieces of manganese dioxide in the cleansed flask (fig. 36). Pour in strong hydrochloric acid which has been mixed with about one-third its measure of water, and heat the mixture gently in a draught closet. A greenish-yellow gas will be evolved. Collect the gas by displacement (Exp. 16, 37), since it is much heavier than air ·

$$MnO_2+4HCl=Cl_2+MnCl_2+2H_2O.$$

This gas has a very destructive action on the lungs, and must on no account be inhaled in any quantity.

60. **Tests for Chlorine.**—Chlorine is usually recognised by its yellowish-greenish colour, by its peculiar smell, and by its property of bleaching moist vegetable colours.

EXPERIMENT 44 —The bleaching-power of chlorine is shown by placing a piece of moistened litmus-paper, or of *Turkey-red* fabric dyed with madder, in a jar of the gas. The colour will be destroyed.

When a burning candle is plunged into a jar of chlorine, it continues to burn with a very smoky flame. A strip of filter-paper wetted with hot turpentine catches fire spontaneously in chlorine, and gives rise to dense smoke. The hydrogen only of these combustible substances combines with the chlorine forming hydrogen chloride, and the carbon separates in the free state giving rise to the smoke.

A piece of dutch-foil or of copper-leaf also burns spontaneously when it is dropped into chlorine, the metal being converted into its chloride with production of heat.

If chlorine is allowed to bubble through a little water, the water will acquire the colour, the smell, and the bleaching-power of the gas. This shows that chlorine is soluble in water, and explains why the gas cannot be collected over water without loss.

When chlorine is brought into contact with hydrogen sulphide (fig. 34, p. 45), hydrogen chloride is formed and sulphur is deposited on the inside of the vessel.

HYDROGEN CHLORIDE, OR HYDROCHLORIC ACID GAS.

61. Preparation of Hydrogen chloride—When sodium chloride and sulphuric acid are heated together in a glass vessel, they furnish hydrogen chloride and sodium hydrogen sulphate:

$$NaCl + H_2SO_4 = HCl + NaHSO_4.$$

EXPERIMENT 45.—Cleanse the flask which was used for the preparation of chlorine (fig. 36, 59), and place in it several lumps of rock salt (sodium chloride). Pour in some strong sulphuric acid which has been diluted with an equal volume of water, and heat gently. Hydrochloric acid gas will be evolved. Its escape will be rendered evident by the fumes which it produces in ordinary moist air and by its pungent smell, also by its power of turning blue litmus-paper red. This gas is heavier than air and should be collected by displacement (fig. 36), since it dissolves largely in water.

62. Tests for Hydrogen chloride.—This gas may be recognised by its pungent smell, by fuming in the air, by turning blue litmus red, and by giving opaque white fumes with ammonia gas or with strong ammonia solution (53). A still more characteristic test depends upon its power of rendering silver nitrate solution turbid by the formation of insoluble silver chloride.

EXPERIMENT 46.—Introduce some silver nitrate solution, which has been acidified with nitric acid, into the gas: the liquid will at once become turbid. The experiment may be tried by pouring some of the silver solution into a jar containing the gas; by allowing the gas to pass into the solution; or by dipping a glass rod into the silver solution, and then introducing the rod, with a drop hanging upon its end, into a jar of the gas.

SULPHUR DIOXIDE, OR SULPHUROUS OXIDE GAS.

63. **Preparation of Sulphur dioxide from Sulphuric acid.**—This gas has already been made by burning sulphur in oxygen (24, Exp. 6). It may also be prepared from strong sulphuric acid by heating this acid with copper.

EXPERIMENT 47.—Cleanse the preparation flask (fig. 36, p. 49), and place some copper turnings in it. Then pour in strong commercial sulphuric acid and heat the mixture. Sulphurous oxide will be evolved when the acid has been heated nearly to boiling:

$$Cu + 2H_2SO_4 = SO_2 + CuSO_4 + 2H_2O.$$

The gas may be cooled and washed by passing it through *a little* water. It is then collected by displacement, since it is much heavier than air and is extremely soluble in water.

64. **Tests for Sulphur dioxide.**—This gas possesses an extremely pungent smell, and is identified by the following tests, of which the first is the most characteristic.

EXPERIMENT 48.—Pour into a jar of the gas some dilute solution of potassium dichromate and shake it round; also hang paper moistened with the dichromate solution in the jar: the reddish

Page 51, par. 65, should read as follows:

65. **Preparation of Nitrogen monoxide.**—When solid ammonium nitrate is heated, it melts; and when it is further heated, it decomposes into steam and nitrogen monoxide: $NH_4NO_3 = N_2O + 2H_2O$. Care must be taken to regulate the heat as soon as the effervescence of the melted salt commences, else the nitrogen monoxide gas and steam may be evolved with dangerous rapidity.

EXPERIMENT 49.—Cleanse the flask (fig. 36, 59), and close it with a singly perforated cork bearing a delivery-tube which has been bent twice at right angles. Replace the washing-bottle by a flask which is closed by a doubly perforated cork, bearing tubes which pass just through the cork; and weight the flask by pouring shot into it, so that it may be sunk into a pan of cold water and serve to condense the steam which escapes with the gas, and to cool the gas. Now heat the nitrate in the flask; and collect the nitrogen monoxide, which is much heavier than air, by displacement.

66. Tests for Nitrous oxide.—This gas has a sweet taste; it is detected by the "spark-test," and by not colouring nitric oxide.

EXPERIMENT 50.—Show that nitrous oxide kindles a spark on a splinter of wood into flame, but that it differs from oxygen by producing a greenish halo around the flame. Show further that nitrous oxide gives no red colour with nitric oxide, as oxygen does (25, 44) for this experiment the gas should be collected over water, since it may contain air if it has been collected by displacement.

ETHENE, OR ETHYLENE GAS.

67. Preparation of Ethene from Alcohol.—Ethene may be prepared by heating alcohol with strong sulphuric acid:

$$C_2H_6O + H_2SO_4 = C_2H_4 + H_2SO_4\ H_2O.$$

EXPERIMENT 51.—The apparatus shown in fig. 35 (55) may be used. The flask should, however, be round and not conical in shape, and must not be less than fourteen ounces in capacity, since the preparation-mixture froths considerably.

A mixture of one volume of alcohol, of 0·83 specific gravity, with five volumes of strong sulphuric acid, is made by shaking the liquids together in the generating-flask. This mixture must not be more than half an inch in depth in the flask. A solution of sodium hydrate is placed in the two-necked washing-bottle, and serves to absorb the sulphur dioxide which escapes during the preparation.

The mixture in the generating-flask is heated carefully with constant watching, since it is apt to froth.

Ethene when mixed with air may burn explosively, and care must therefore be taken to expel the air from the preparation vessels completely before any quantity of the gas is collected and burnt. Two cylinders should be filled with the gas by collecting it over water, and another dry jar should be filled by direct displacement of the air.

68. Tests for Ethene.—Ethene may be recognised by the character and products of its flame, and by its combination with bromine.

EXPERIMENT 52.—Burn the ethene in one of the wet jars and note its very luminous flame. Then burn the gas contained in the dry jar and close the jar with a glass plate as soon as the combustion is ended: note that the inside of the jar has become dimmed with moisture: now shake up the products of combustion with lime-water, the lime-water will become milky proving that carbon dioxide is also a product of the combustion of ethene

Open the second wet jar containing ethene for a moment, pour into it a little bromine-water and shake the liquid with the gas, The colour of the bromine will disappear and a colourless fragrant liquid, ethene dibromide, will be formed

This power of decolourising bromine is a characteristic property of ethene and of certain allied gases.

METHANE GAS, OR MARSH GAS.

69. **Preparation of Methane from Sodium acetate.**—This gas may be prepared by heating an intimate mixture of dried sodium acetate with soda-lime:

$$NaC_2H_3O_2 + NaOH = CH_4 + Na_2CO_3.$$

EXPERIMENT 53.—Heat some crystallised sodium acetate in a shallow iron sand-bath over the Bunsen-flame, until steam is no longer given off. The salt will set into a white mass as it cools. Mix one part of this dry salt with four parts of dry soda-lime, then reduce the mixture to fine powder, and heat the powder to redness in an apparatus such as that shown in fig. 21 (p. 22). It is necessary to substitute a hard-glass tube for the test-tube, since an ordinary test-tube would be softened by the heat which must be applied for the above decomposition. As soon as the air has been expelled from the apparatus, fill two cylinders with methane over water in the usual way, and fill another dry cylinder by direct displacement of the air by the light gas.

70 **Tests for Methane.**—Repeat with this gas the tests which have been described under ethene (68, Exp. 52). The flame of methane will be found to be less luminous than that of ethene but it will yield the same products. Bromine water will not lose its colour when it is shaken with the gas, showing that bromine does not combine with methane.

ACETYLENE.

71. **Preparation of Acetylene from Calcium carbide**—When calcium carbide is dropped into water, acetylene gas is rapidly given off:

$$CaC_2 + H_2O = C_2H_2 + CaO.$$

EXPERIMENT 54.—Fill a small cylinder or a boiling tube with water; close the mouth and invert it, mouth downwards, in a vessel of water, so as to retain the water in the cylinder. Drop into the vessel a piece of calcium carbide about the size of a hazel-nut, and collect the gas by shifting the tube until its mouth is over the piece of calcium carbide.

When the tube is full of gas remove it from the water and ignite the gas. It will burn with a luminous and very smoky flame.

EXPERIMENT 55.—This gas may be prepared in larger quantities by an apparatus similar to that shown in fig. 24 (p. 28). The calcium carbide is placed in the dry flask fitted with a dropping funnel containing water and a delivery-tube for gas. When the water is allowed to drop slowly on the carbide, a rapid evolution of acetylene takes place. The gas may be collected over water in the usual way.

72. **Properties and Tests.**—Notice that acetylene as thus prepared is a colourless gas, and possesses an offensive smell which is due to the presence of impurities Prove that it burns with a very luminous and smoky flame. Also that it forms a white precipitate when it is brought into contact with ammoniacal solution of silver nitrate and a red precipitate of cuprous acetylide with ammoniacal cuprous chloride The formation of the copper precipitate is a delicate test for acetylene.

II.—DETECTION OF GASES.

73. **Certain Properties of the Gases**, which have been already described, enable these gases to be detected when they occur alone and frequently when they are in a state of mixture. These properties, which serve as tests for the presence of each gas, are stated below, and are given in greater detail under the preceding descriptions of the individual gases.

The student may proceed to apply these tests to the detection of gases occurring singly and in a state of mixture.

When a mixture of gases is being examined, it should be remembered that those which act chemically upon one another cannot be simultaneously present. Such mutually exclusive pairs of gases are —O and NO ; HCl and NH_3, SO_2 and H_2S ; Cl and H_2S , NH_3 and SO_2 ; H and Cl in daylight.

In testing for the constituents of a mixture, a brief exposure of a part of the mixture to air would detect NO; shaking another portion with water, followed by examination of separate portions of this solution, would detect HCl, NH_3, SO_2, H_2S, Cl, CO_2; application of a flame to another portion of the mixture would detect O, H, N, H_2S, CO, CH_4, C_2H_4 and C_2H_2 Special confirmatory tests should be applied, when possible, to support the conclusions drawn from the above general tests.

Some gases are difficult to detect when certain other gases are mingled with them in large proportion. Advantage may frequently be taken of their solubility in water or in solution of caustic alkali to remove these interfering gases and to facilitate the detection of insoluble gases.

Oxygen: kindles a glowing splinter into flame, and gives reddish-brown gas with nitric oxide (25), it is not soluble in water.

Nitrogen: is incombustible, extinguishes a flame (28), and gives negative results with all the tests which are mentioned for other gases.

Hydrogen: burns with a non-luminous flame, which deposits moisture and forms no carbon dioxide (36), it is not soluble in water.

Nitrogen monoxide: is colourless; is unchanged by contact with air; has a sweetish taste; does not colour nitric oxide; kindles a glowing splinter into flame, producing a pale yellowish green halo around the flame.

Nitric oxide: is colourless, but becomes reddish-brown when it is mixed with air or with oxygen (44).

Carbon dioxide: extinguishes flame; is incombustible; makes lime-water milky; dissolves slightly in water, and the solution renders lime-water milky (42).

Hydrogen sulphide: possesses a foul smell; burns with a blue flame, yielding water and sulphur dioxide; dissolves in water; the aqueous solution blackens a drop of lead acetate solution (48).

Ammonia: possesses a pungent smell; gives dense white fumes with hydrogen chloride gas (53); dissolves in water; the aqueous solution turns red litmus blue and turmeric brown.

Carbon monoxide: burns with a blue flame, forming no water; after the combustion carbon dioxide is found by shaking the product with lime-water (57); is not soluble in water.

Chlorine: possesses a peculiar smell, and causes coughing; a taper burns in the gas with a red and smoky flame; dissolves slightly in water; the aqueous solution bleaches litmus (60); both the gas and its solution turn paper, soaked in solution of starch and potassium iodide, blue.

Hydrogen chloride: fumes in the air; gives dense white fumes with ammonia gas; dissolves in water; the aqueous solution reddens blue litmus, and gives a white curdy precipitate when it is mixed with silver nitrate solution (62).

Sulphur dioxide: possesses a pungent smell, dissolves in water, the aqueous solution turns blue litmus red, and changes the red colour of a drop of potassium dichromate solution to green (64).

Methane: is free from smell, burns with a slightly luminous flame, producing water and carbon dioxide; does not decolourise bromine water (70) · is not soluble in water.

Ethene: has very faint smell; burns with a very luminous and somewhat smoky flame, producing water and carbon dioxide; decolourises bromine water (68); is slightly soluble in water.

Acetylene: has an offensive smell as ordinarily prepared; burns with very luminous and smoky flame, producing water and carbon dioxide; produces with ammoniacal cuprous chloride solution a red precipitate (72).

SECTION III.

ANALYTICAL OPERATIONS.

Introductory Remarks.—Before the student tries the analytical reactions, he should become familiar with the operations which are commonly employed in chemical analysis. The processes will be easily understood by reading through the following descriptions, and then performing the illustiative experiments.

Caution.—In all analytical work the *water* used must be *distilled water* (81, 82, 83), and this only should be kept in the wash-bottle.

DISTILLATION, DISTILLED WATER.

81. **The Process of Distillation** is often used to purify a liquid from solid substances which it contains in solution (84). The process consists in boiling the liquid and cooling or *condensing* the vapour. The solid substances are left behind in the vessel in which the liquid is boiled, and the condensed vapour forms the liquid *distillate.*

The purification of spring water from the salts dissolved in it is an example of this process.

82. **Tests for Salts dissolved in Tap-water.**—Certain salts which are ordinarily present in spring water may be tested for as is directed below. But it must be understood that all the results which are described may not be obtained, since the soft water supplies of some of our large towns are almost free from dissolved solids. Rain water or soft water which gives no results with these tests may be employed in qualitative analysis without previous distillation. A sample of water which contains saline impurities must be obtained for the following tests.

EXPERIMENT 59.—Make each of the following tests on a separate portion of the water contained in a test-tube.

Add to some of the water several drops of dilute nitric acid and of silver nitrate solution, and notice whether the water becomes turbid. Turbidity shows the presence of *chloride* in the water.

A separate portion of water in another test-tube may be found to become turbid on the addition of some solution of ammonia and of ammonium oxalate. Dissolved *calcium* salts produce this turbidity.

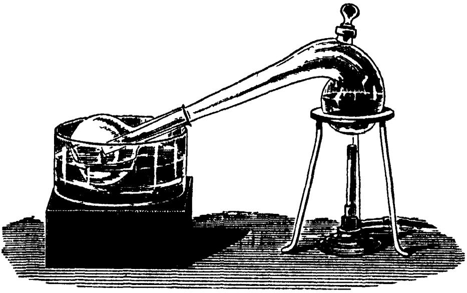
The presence of *sulphate* would be indicated by a turbidity being caused by the addition to another portion of the water of a few drops of hydrochloric acid and of barium chloride solution.

Ascertain also whether precipitation of *soap* is caused by the water, owing to the presence in it of certain dissolved solids. For this purpose dissolve a small shaving of soap by warming it in a little distilled water. Add a drop of this soap-solution to some of the water, which half fills a test-tube. Close the mouth of the test-tube with the thumb, and shake the water well. If *hardening* salts are present no lather will form, but the water will be rendered more or less turbid by the separation of soap. By the gradual addition of more soap-solution, the initial turbidity may be increased, and a lather may be produced when the water is shaken.

83. Tests for the Purity of Distilled water.—Distilled water is made in large quantities for use in various chemical processes. It must be substituted for ordinary water in the process of analysis, if the water-supply contains dissolved solids. The student may use a glass retort and cooled flask (fig. 37) to produce a little distilled water for the following tests, or he may use some of the distilled water supplied to the laboratory. An apparatus for preparing distilled water in large quantity is described in Section VII.

EXPERIMENT 61.—Distilled water when examined by the tests described in paragraph 82 should remain perfectly clear. Solution of ammonium oxalate, of silver nitrate, of barium chloride, or of ammonium sulphide when separately added to a fresh portion of the water should produce no precipitate. These tests prove the absence of calcium salts, of chloride, of sulphate, and of lead and iron salts respectively, any or all of which have been removed by distillation. Prove also that distilled water at once gives a lather with soap-solution, and leaves no residue when it is evaporated to dryness (87).

FIG. 37.



DISTILLATION OF WATER.

SOLUTION.

84. The Process of Solution.—Many solid substances gradually *dissolve* in water, when they are stirred or shaken with that liquid. Salt and alum may be mentioned as examples. Certain other liquids may be employed instead of water; and if they cause solid substances which are immersed in them to become partially or entirely liquid, and to mingle uniformly with the liquid, they are said to *dissolve* the solids.

The liquid thus obtained is called a *solution* of the solid, and the liquid which dissolves the solid is termed the *solvent.*

Further, a solid which dissolves in a liquid is said to be *soluble* in that liquid; if it does not dissolve, it is said to be *insoluble.*

Thus when water is shaken with sodium chloride or common salt it dissolves the salt, yielding solution of sodium chloride; water is therefore called a solvent for sodium chloride, and this salt is said to be soluble in water.

The process of solution proceeds most rapidly when the solid substance is finely powdered before it is stirred in the liquid, since the largest possible surface of the substance is thus brought into contact with the solvent.

The process is also much hastened *by heating the solvent*, partly because heat causes a rapid circulation of the liquid over the solid, and also because a solid substance is usually much more soluble in the hot liquid than in the cold one.

Two kinds of solution may be distinguished, namely *simple solution* and *chemical solution.*

85. **Simple Solution** occurs when a substance is dissolved by a liquid without undergoing alteration in its composition. A simple solution generally possesses the taste, colour, and other general properties of the solid which it contains. It also yields the solid substance again when the solvent is removed by *evaporation.* The solution of potassium nitrate or of copper sulphate in water is an example of a simple solution.

EXPERIMENT 63.—Place a piece of potassium nitrate in a small clean beaker. Partly fill the beaker with water, and stir the solid about with a glass rod. The potassium nitrate will *slowly* dissolve in the water, and the solid will diminish in amount.

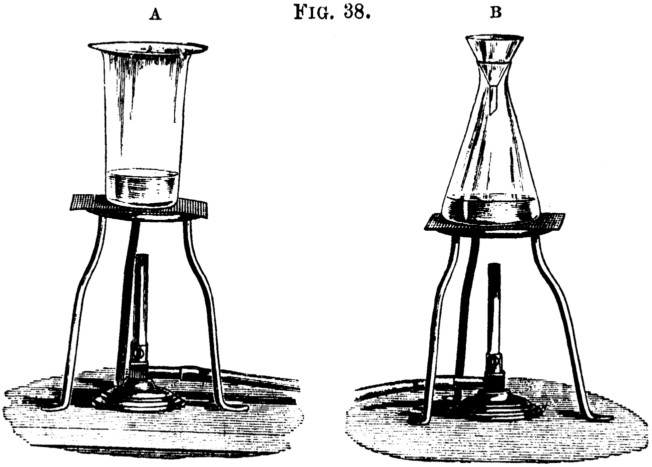
Now heat the liquid by placing the beaker on wire-gauze over a small flame; the solution will proceed much more rapidly.

Powder another piece of potassium nitrate by crushing it in a mortar, and then rubbing it round with the pestle. Place this powder in a beaker, pour in water, and heat the bottom of the beaker on wire-gauze by a small Bunsen-flame (fig. 38A). The potassium nitrate will dissolve much more rapidly than before, showing that the process of solution is accelerated by powdering the solid and by employing heat. Mix these solutions, and preserve the liquid for future use.

The forms of apparatus which are used for dissolving substances in larger quantities are represented in figs. 38 A, B. Two devices for preventing the loss of the boiling liquid by spirting, and for checking loss by evaporation, are seen in these figures. The beaker is covered with a clock-glass, and the neck of the flask is partly covered by inserting a funnel. These precautions against loss are not usually necessary.

Experiment 64.—Powder a little copper sulphate in a mortar, and transfer it to a small porcelain dish. Half fill the dish with water, support it upon a pipe-clay triangle placed on a tripod or retort-stand, and heat it with a small flame. The *blue* copper sulphate will dissolve, yielding a *blue* solution. Keep this solution for future use.

Fig. 38.



Apparatus for Solution of a Solid.

These experiments (63, 64) are examples of simple solution. The first shows that a colourless solid gives a colourless solution, and the second that a coloured solid gives a coloured solution. This is generally true, and hence the presence of a coloured substance in a solution may be inferred if the liquid itself is coloured.

Moreover if a drop of the potassium nitrate solution is tasted, it will be found to possess the same taste as the solid.

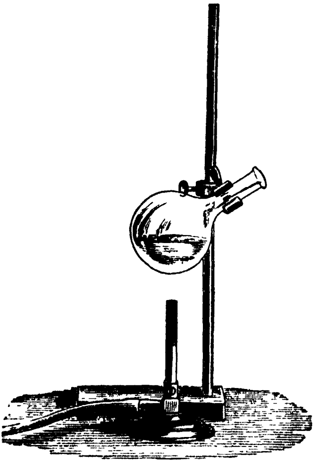
When a *simple solution* is being analysed its colour is usually noted, and its taste is occasionally tried with proper precautions.

86. Chemical Solution differs from simple solution by producing a chemical change in the substance which is undergoing solution. The solution therefore contains a substance which differs in composition from the undissolved solid. Accordingly when the solvent is removed by evaporation the original substance is not obtained.

Experiment 65.—Place in a test-tube a small piece of marble or of calcium carbonate, pour upon it a little water and heat the tube. The calcium carbonate will be found to be insoluble in water.

Now add to the water some hydrochloric acid: *effervescence*, or escape of numerous small bubbles of gas, will occur. The piece of calcium carbonate will meanwhile slowly diminish in size and will at last entirely disappear in the liquid, if sufficient acid is added.

Fig. 39.



Solution of a Solid.

Experiment 66.—Place in a test-tube a small piece of copper, and warm it with a little water: the copper will remain undissolved. Now add to the water some nitric acid and heat: the copper will slowly dissolve, yielding a reddish-brown gas. The metal may be entirely dissolved, if sufficient nitric acid is employed.

These are two examples of chemical solution. The calcium carbonate is changed by the hydrochloric acid into calcium chloride, and this substance, *not the calcium carbonate*, remains in solution. The copper is changed into copper nitrate, which is then dissolved by the water. These solutions would therefore furnish on evaporation calcium chloride and copper nitrate respectively, and not marble or metallic copper.

It will be noticed that in each of these cases a gas is given off. This is a very usual, but not a universal effect of the process of chemical solution. The distinguishing fact is that the solid substance has undergone a change in composition in the act of passing into solution.

Usual methods of effecting chemical solution are shown in figs. 38 and 39.

It is sometimes necessary to dissolve a substance without exposing it to contact with the air during the process. This may be effected by passing a stream of carbon dioxide through the vessel in which the solution is proceeding; or the air may be displaced from the flask by carbon dioxide, and then a perforated cork may be at once fitted into the neck of the flask. The perforation carries either a bent tube with its end just immersed in a small quantity of distilled water (fig. 40), or a short tube surmounted by a piece of rubber tube, which is closed with a solid glass stopper: the rubber tube has a slit cut in it by a sharp knife (fig. 41), and this slit gives egress to any gases from within, but excludes the entrance of air from without.

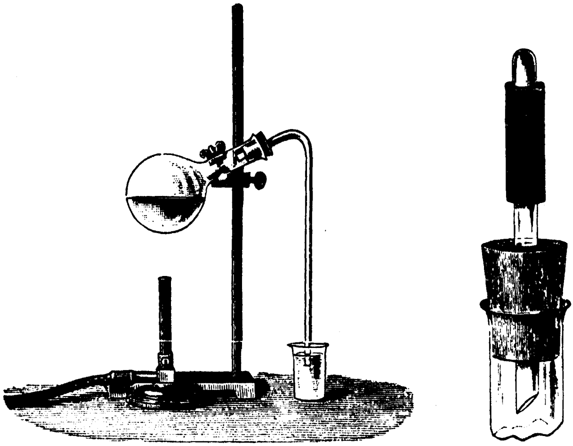


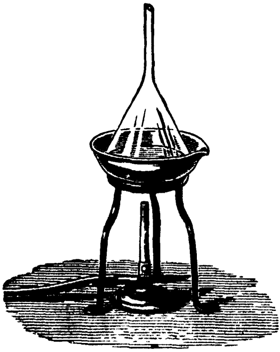
FIG. 40. FIG. 41.

SOLUTION OF SOLID OUT OF CONTACT WITH AIR.

EVAPORATION AND CRYSTALLISATION.

87. The Process of Evaporation.—When it is necessary to separate a solid substance from the liquid in which it is dissolved, the liquid is boiled away as vapour or *evaporated.* The solid substance is then left behind in the vessel. The liquid is usually evaporated by heating the solution in an open porcelain evaporating dish over the Bunsen-flame.

FIG. 42.



EVAPORATION IN DISH.

EXPERIMENT 67.—Pour the potassium nitrate solution (Exp. 63) into a porcelain evaporating basin, and heat it over the Bunsen-flame until the water has been nearly boiled away. Then make the flame smaller, and continue heating until the water has disappeared. Solid potassium nitrate will be left in the dish.

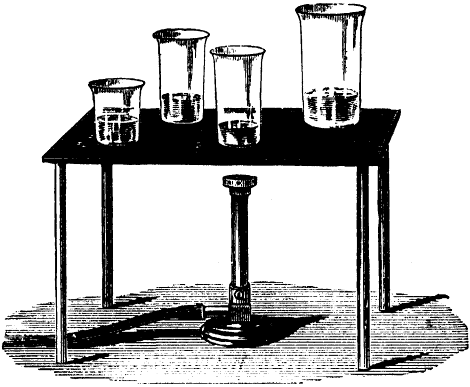
Towards the end of the process of evaporating a solution, a small flame should always be used: and in order to prevent the substance from spirting out of the dish, the flame is moved about, or the dish may be covered with a funnel (fig. 42) or with a round filter-paper.

The process of evaporation over a naked flame is shown in fig. 42, and the covering of the liquid during the latter stage of the process is indicated. Fig. 43 illustrates another method of evaporating on a heated iron plate, the liquid being contained in a beaker instead of in a dish: the nearer the beaker is placed to the part of the plate which is over the flame, the more rapid will be the evaporation.

88. Evaporation at Steam-heat.—It is often necessary to evaporate more slowly at steam-heat, in order to prevent the spirting of the liquid or the over-heating of the solid. For this purpose the dish is placed over boiling water upon a water-bath, steam being in this case the heating agent.

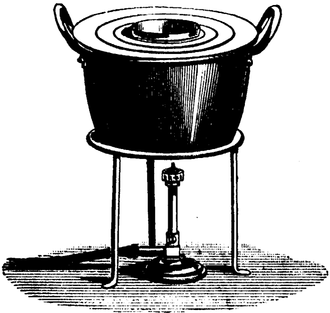
A simple form of water-bath is shown in fig. 44. It consists of a copper vessel which is rather more than half-filled with water, and is heated by a Bunsen-burner. The upper portion is fitted with flat sheet-copper rings of gradually diminishing diameters, and the top of the bath can thus be made to support vessels of various sizes.

FIG. 43.



EVAPORATION IN BEAKERS.

FIG. 44.

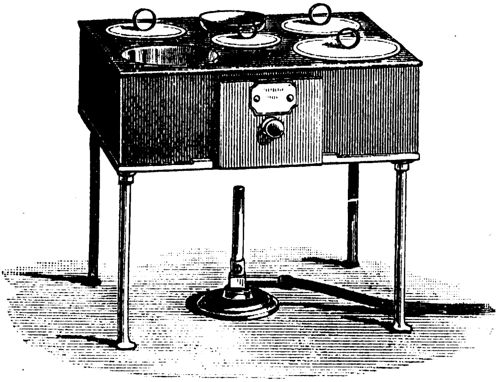


SINGLE WATER-BATH.

A more complex form of water-bath is shown in fig. 45. In the upper surface of this bath there are holes of various sizes, designed to fit vessels of different dimensions. When these holes are not in use, they are covered by lids as is shown in the figure. This water-bath may be fitted with a constant water supply by the automatic arrangement represented in fig. 58 (98).

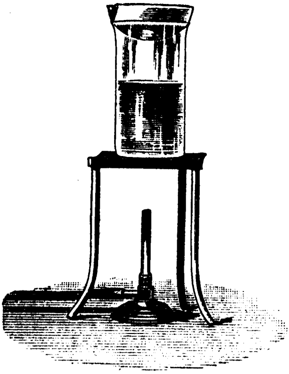
A simple water-bath may be improvised from a beaker partially filled with water and heated by a Bunsen-flame (fig. 46). A few small pieces of paper may be thrown into the water in order to prevent it from bumping during the boiling.

FIG. 45.



MULTIPLE WATER-BATH.

FIG. 46.



SIMPLE FORM OF WATER-BATH.

When the evaporating dish is placed on the top of the beaker, it may possibly prevent the escape of the steam. If this should be the case, some strips of paper may be inserted between the bottom of the dish and the edge of the beaker.

89. Concentration and Crystallisation.—Sometimes only a part of the liquid is evaporated for the purpose of *concentrating* the solution. When a hot solution has been sufficiently concentrated, it will frequently deposit *crystals* of the dissolved substance as it cools, Soluble substances are frequently dissolved, and then crystallised from their solution in this way, in order to free them from impurities.

Experiment 68.—Concentrate the copper sulphate solution which was prepared in Exp. 64, and allow the liquid to cool. If sufficient water has been evaporated, crystals will form. Keep the copper sulphate in the dish for future use.

Precipitation.

90. Precipitation by a Liquid.—When two clear and transparent solutions are mixed together, they frequently become more or less turbid or opaque, owing to particles of a solid substance being formed in the liquid. A solid substance thus produced is called a *precipitate,* and the process of producing a precipitate is termed *precipitation.*

A substance is frequently removed from solution by causing it to form an insoluble compound or precipitate. The substances which are added for this purpose are termed *reagents* or *precipitants :* they are commonly liquids.

In producing a precipitate, care must be taken that the two solutions are well mixed. Mixture may be effected by warming the bottom of the test-tube in the flame, by stirring the liquid with a glass rod, or by pouring the liquid from one vessel to another.

A precipitate often appears more rapidly when the liquid is vigorously shaken, or when it is warmed.

Experiment 69.—Pour some barium chloride solution into a test-tube, and add ammonium carbonate solution. A white precipitate of barium carbonate will form. Keep the precipitate and liquid in the test-tube for future use.

In this instance barium chloride and ammonium carbonate may be readily dissolved in separate portions of water. But if these solutions are mixed, two different substances, ammonium chloride and barium carbonate, are produced.

The former of these remains dissolved in the water; but the latter is insoluble, and therefore separates as a fine powder in the liquid.

Precipitates differ much in colour and in general appearance and properties; they are therefore frequently produced, by the addition of suitable reagents, in order to show the presence of a substance. Precipitates are also produced for the purpose of separating one substance from another.

The appearance of a precipitate is usually described by its colour and by its condition. It is *flocculent*, if it forms in flock-like masses; *crystalline*, if it consists of small particles which are seen to be crystals under a lens or microscope; *gelatinous*, if it is jelly-like in its consistency. The formation of a slight precipitate may cause only a *turbidity* in a liquid.

The colour of a precipitate is often falsified by gaslight. If the colour is to be seen at night, it should be examined by the light produced by the electric arc, or by a piece of burning magnesium ribbon.

91. **Precipitation by a Solid Reagent** is occasionally resorted to. Thus a metal is not unfrequently precipitated from the solution of its salt by the immersion in it of another metal.

EXPERIMENT 70.—Dip a clean penknife-blade into some of the copper sulphate solution (Exp. 68), to which a few drops of sulphuric acid have been added. After a short time metallic copper will be precipitated from the solution and covers the iron as a red film.

FILTRATION.

The Processes of Filtration and of Decantation (94) serve to separate a precipitate from a liquid in which it is suspended.

92. **Filtration.**—The liquid containing the precipitate is

poured upon the porous paper. The liquid itself runs through the pores of the paper, but the solid particles of the precipitate are retained upon the surface of the paper.

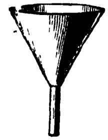
The paper employed is called *filter-paper*. The liquid which has passed through the filter is termed the *filtrate*. A filtrate may frequently be coloured by some substance *dissolved* in it, but it should be perfectly free from turbidity caused by solid particles *suspended* in it.

The ordinary process of filtration, as it is employed in chemical analysis, is described below : methods of filtration on a larger scale are described in paragraphs 1144, 1145.

Prepare a filter for filtration as is directed below.

EXPERIMENT 71.—Measure a glass funnel (fig. 47) along its sloping side from shoulder to rim. Select a circular filter-paper, the radius of which is somewhat less than this in length: fold it across into a semicircle, then fold it again at right angles into a quarter of a circle; now open out either of the sides, so as to form a little conical bag (*d*, fig. 48). This forms an ordinary filter.

FIG. 47.



FUNNEL.

If ready-cut filters of suitable size are not at hand, cut from a sheet of filter-paper a square piece, the edge of which is rather less than double the length of the side of the funnel. Fold it over along the dotted line (*a*, fig. 48) ; then again along the dotted line (*b*). This gives a square (*c*) which at one angle has four free corners ; these are removed by cutting with a pair of scissors along the curved dotted line shown in (*c*).

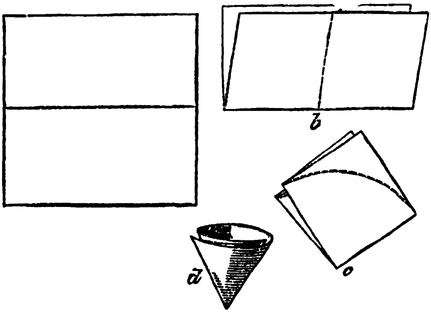
The *filter* is now made and only requires to be opened. By separating the curved edges so that they form a circle, three remaining on one side of the circle and one on the other, a little closed pointed paper bag is formed (*d*).

A filter of the same shape, but of uniform thickness, may be made from half an ordinary circular filter-paper, or from half of the circular flat paper formed by opening out the filter (*d*). This semicircular paper is folded into a quadrant shape, and the two radial edges are doubled over together several times, the fold being pressed down with the finger nail. The unused half of the filter (fig. 48. *d*) is absent in the filter which is formed by opening out this paper, and filtration is more rapid in consequence.

The folded filter is now gently pressed *with dry fingers* into the *dry* funnel, and the folding is altered if necessary until the paper fits the glass closely. It is then moistened all over with water from the wash-bottle and is ready for use.

This preliminary moistening of the filter-paper must not be neglected; since if the liquid which contains the precipitate is poured upon a dry filter, some particles of the precipitate may get between the fibres of the paper; the paper then shrinks on being wetted, and thus incloses the solid particles which choke the pores of the filter. Filtration may in this way be seriously retarded.

FIG. 48.



FOLDING AND CUTTING FILTER.

During the filtration of a liquid the funnel may be placed with its neck in a test-tube, which is supported in the test-tube stand. Care must, however, be taken that the inside of the upper part of the test-tube is dry, and that there is a space between the neck of the funnel and the inside of the tube; since, if the egress of air from the test-tube is prevented, the filtration will be stopped.

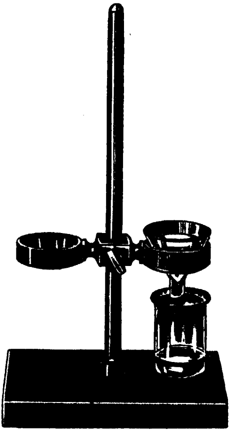
The funnel is therefore preferably supported upon the ring of a wooden filter-stand (fig. 49); and the filtrate is received in a small beaker standing beneath the funnel, instead of in a test-tube.

As has been already stated, the upper edge of the filter must always be below the rim of the funnel: and when the liquid, which is to be filtered, is being poured into the filter, great care must be taken not to let it reach the upper edge of the filter-paper.

The precipitate should not more than half fill the filter, else it cannot conveniently be washed.

Experiment 72.—Pour the liquid containing the precipitate of barium carbonate, from Exp. 69, through the filter. If the above directions have been carried out, and no hole has been made in the paper, while it is being prepared or fitted into the funnel, the filtrate will run through perfectly clear, leaving the barium carbonate on the filter. Keep the filter and precipitate in the funnel for future use.

Fig. 49.



Filter-Stand.

Sometimes the filtrate passes away from the filter in a turbid condition. In order to remove this turbidity, the turbid portion of the filtrate must be poured once or twice through the same filter. Or the turbidity may be prevented by pouring the liquid at first through a double filter, which is prepared by folding two filter-papers together as one : a double filtration is thus secured in one operation.

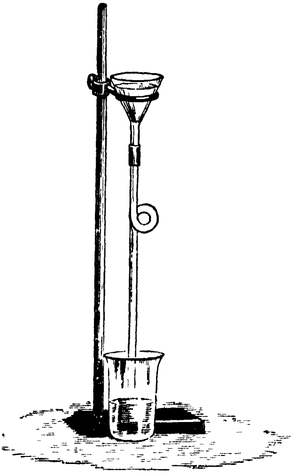
It should be borne in mind that a hot liquid passes through the filter more quickly than a cold one. Hence, if it is not inadmissible for other reasons, a solution should always be heated before it is filtered.

Common filter-paper may contain a little calcium carbonate. This is of no importance in an ordinary analysis, and is only objectionable when an acid solution has to be examined for traces of calcium after filtration. In order to remove this impurity, the filter is fitted into the funnel in the usual way and is moistened with dilute hydrochloric acid; it is then well washed with distilled water.

This process of treatment is, however, seldom necessary, since filters may be obtained at small cost, which are practically free from all matter which is soluble in acids.

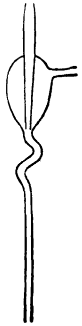
93. **Accelerated Filtration** —Methods for expediting the filtration of large quantities of solids from liquids are described in paragraph 1143. But it is often desirable to hasten the process of filtration of small quantities of solid from a liquid in any ordinary funnel, more especially when a flocculent precipitate is being separated from a liquid. This may be effected either by attaching a suitable tube to the funnel, or by means of a filter-pump or an aspirator.

FIG. 50.



FILTER-TUBE.

FIG. 51.



GEISSLER-ASPIRATOR.

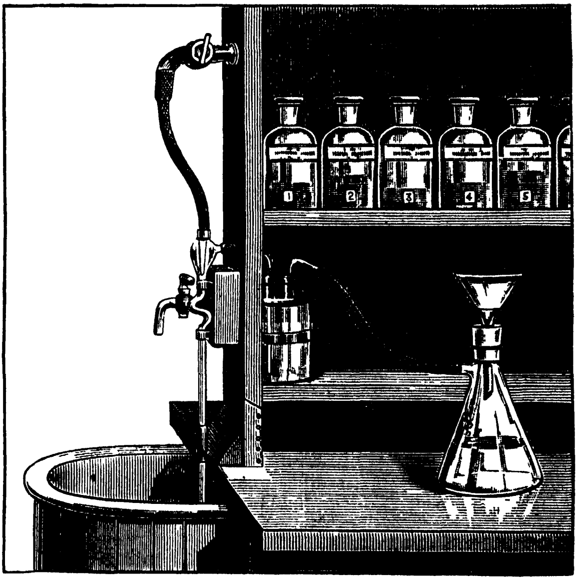
(1) *One of the simplest means* of increasing the rate of filtration is to attach to the funnel a glass tube, bent as is shown in fig. 50. As soon as the filtrate has filled this tube, the weight of the column of liquid tends to draw the liquid through the filter, and the rate of filtration is accordingly increased.

Another simple method is to attach to the funnel a *straight* piece of glass tube about 2 feet in length and 1 millimetre in bore. The liquid column which is suspended in this tube acts in the way just described.

(2) *A Filter-pump, or Aspirator*, which applies suction to the neck of the funnel, is frequently useful to hasten the process of filtration.

The accompanying illustration (fig. 52) shows a convenient arrangement for this purpose. The glass Geissler-aspirator (fig. 51) is shown over the sink, connected by stout rubber tubing, inclosing canvas, with the high pressure water-tap. When the water is turned on and flows through the aspirator, air is sucked from the thick-glass conical tubulated filter-flask; and the atmospheric pressure, acting on the liquid in the funnel, is thus utilised for forcing the liquid through the filter.

FIG. 52.



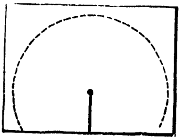
WATER FILTER-PUMP, OR ASPIRATOR.

The two-necked Woulffe's bottle is interposed between the aspirator and the filter-flask in order to prevent the water in the aspirator from entering the flask.

The neck of the funnel is fitted by a rubber cork into the neck of the conical filter-flask; and in order that the pressure which is exerted on the point of the filter-paper may not burst it, either toughened filter-paper may be used, or the ordinary filter-paper may be supported beneath by a small filter-cone of muslin or platinum.

The platinum cone is cut from a piece of platinum foil measuring about 1½ × 1¼ inch (fig. 53). A hole is pierced by a pin near the middle of the foil. The foil is cut through from the edge to this hole. The corners are removed by cutting round in a circle which has the pin-hole for its centre. The spring is then taken out of the foil by heating it to redness and cooling it slowly, and the foil is shaped into a cone (fig. 48, *d*, 92), which must be fitted exactly into the bottom of the funnel After the platinum cone has been placed in the funnel, a dry filter-paper is folded as usual and is fitted closely into the dry funnel.

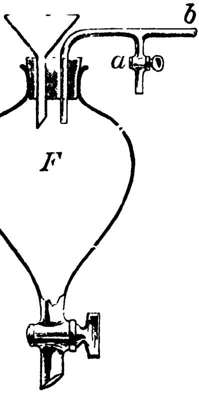
FIG. 53.



PLATINUM FILTER-CONE.

Before the filter is used for filtration it is moistened with water, and is then gently pressed, where necessary, with the fingers until it adheres perfectly to the funnel; the upper edge of the filter especially requires to be carefully pressed against the glass in this way. The filter is now tested, by filling it with distilled water and setting the pump in action. If the paper fits properly and has been pressed into contact with the funnel, no air-bubbles will be sucked down the neck of the funnel until the whole of the water has passed through the filter. Any air-leakage must be stopped by pressing the paper against the funnel where such leakage occurs.

FIG. 54.



SPECIAL RECEIVER FOR THE FILTRATE.

Besides expediting the filtration and washing of precipitates, such an arrangement also enables the precipitate to be dried to a great extent by sucking air through it after the liquid has passed away.

The aspirator may be replaced by other forms of apparatus for producing suction. A common hand-syringe may be used, but this is liable to be attacked by acid vapours if it is made of brass.

A Special Form of Receiver is represented in fig. 54 to serve as a substitute for the conical flask. It consists of an ordinary separating funnel, *F*, which is closed with a doubly perforated rubber stopper. One perforation carries the funnel, which is used for filtration. Into the other is inserted a branched tube, one limb of which (*b*) is connected with the filter-pump, while the other (*a*) can be opened to the atmosphere by turning the stopcock. The use of this receiver renders it possible to withdraw a portion of the filtrate during the process of filtration, by opening simultaneously the stopcocks at *a* and at *c* and stopping the action of the filter-pump.

DECANTATION FROM A PRECIPITATE.

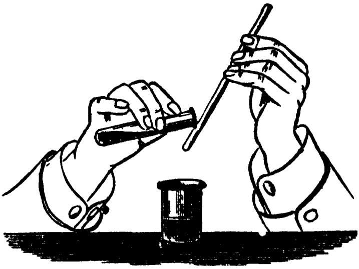
94. **The Process of Decantation** serves to separate precipitates, which settle rapidly from the liquid, without the use of a filter.

For this purpose, the vessel containing the liquid and precipitate is allowed to stand until the precipitate has settled. The liquid is then carefully poured off, or *decanted*, by gently inclining the vessel. A wetted glass rod, pressed against the edge or lip of the vessel (fig. 55), helps to prevent the precipitate from being disturbed while the liquid is flowing away from it.

The process of decantation, if it is carefully performed, will serve to separate the liquid in a clear condition almost completely from a heavy precipitate.

EXPERIMENT 73.—Add *boiling dilute* sulphuric acid to some boiling solution of barium chloride. A heavy precipitate of barium sulphate will be formed. This precipitate may be readily separated from the liquid by decantation. Keep the precipitate in the test-tube for future use.

FIG. 55.



DECANTATION OF A LIQUID.

WASHING THE PRECIPITATE.

95. **The Washing of a Precipitate,** which has been separated from the liquid in which it was suspended, is usually necessary in order to free the precipitate from the adhering solution.

If the precipitate has been filtered, it is washed upon the filter (96); if it has been separated by decantation, it is usually washed by decantation (97).

96. **Washing the Precipitate on the Filter.**—The following directions will serve to explain how this process is carried out, and will indicate what precautions should be observed in order to secure success. The washing process may be hastened by employing the devices which have been already described in par. 93.

EXPERIMENT 74.—Support the funnel, which contains the filter and the barium carbonate precipitate from Exp. 72, in a filter-stand above a beaker or flask (fig. 49, p. 71). Blow into the filter a fine stream of hot distilled water from the wash-bottle (12), so directing the jet as to stir up the precipitate. Fill the paper with water to within a short distance from its edge. Let this water run through perfectly. Then nearly fill the filter again in the manner just described. Repeat this process three or four times, letting the water run through completely each time before adding a fresh quantity.

The precipitate and the filter will probably now be free from everything soluble in water, and the water which passes through the filter will therefore be tasteless. Ascertain whether the washing is complete, by collecting the *last few drops* of the washing-water in a clean test-tube, and testing whether it contains any chloride by adding to the liquid a drop of silver nitrate solution. No turbidity must be produced. If turbidity appears, the washing must be continued, and the washing-water must be tested again. The process of washing must be continued, until silver nitrate produces no turbidity when it is added to the last portions of the water running from the funnel.

97. **Washing the Precipitate by Decantation.**—This method is a quick and easy one with a suitable precipitate.

EXPERIMENT 75.—The precipitate of barium sulphate from Exp. 73 may be washed by decantation as follows.

Hot water is poured into the vessel containing the precipitate, and the water is shaken, stirred, or boiled with the precipitate, which is then allowed to settle. As soon as the water has become clear, it is poured off as completely as possible in the way shown in fig. 55 (94).

By repeating this washing process several times, with the addition of fresh portions of boiling distilled water, the precipitate may be entirely freed from the adhering solution.

While the washing-water is being poured off it should be occasionally tested with blue litmus-paper, in order to ascertain when it ceases to redden the paper and is therefore proved to be free from acid. As soon as the washing-water is quite free from acid, the precipitate may be considered to be thoroughly washed.

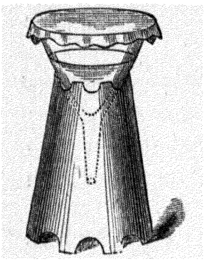
It is usual to Ascertain when the Washing of a Precipitate is Complete, by testing the washing-water for some soluble substance which is being removed by the washing. That substance is usually tested for which is most readily detected. Experiments 74 and 75 serve to illustrate this statement.

DRYING THE PRECIPITATE.

98. **A Precipitate may be dried** by placing the funnel in a hollow tin cone or cylinder, called a *filter-dryer* (fig. 56). The filter-dryer is then either supported on a piece of iron wire-gauze upon a tripod-stand over the flame of a rose-burner turned very low, or it is placed upon a heated iron plate or sand-bath.

The funnel is thus exposed to a current of hot air, which rapidly dries the filter and the precipitate. Great care must be taken so as to regulate the heat as not to char the filter.

FIG. 56.

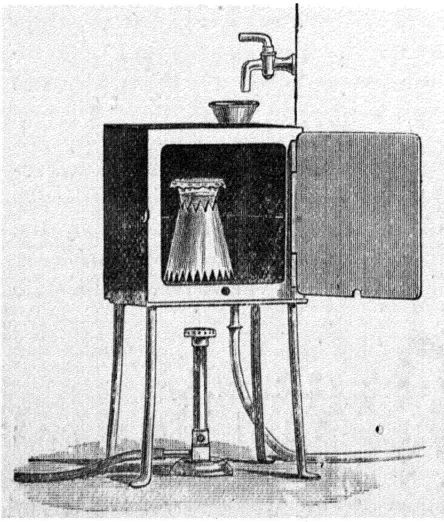


FILTER-DRYER.

The risk of charring the paper may be avoided by placing the funnel, in a perforated shelf, in the steam-oven. This oven is shown in the separate form in fig. 57, and is seen combined with a water-still in fig. 92, par. **1131**.

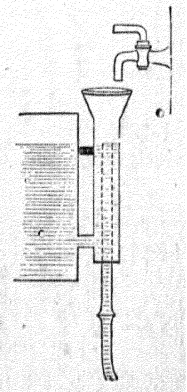
Fig. 58 shows, in section, the arrangement for maintaining the water in the jacket of the steam-oven at a uniform level. A further description of the steam-oven is given in paragraph **1132**.

FIG. 57.



WATER-OVEN.

FIG. 58.

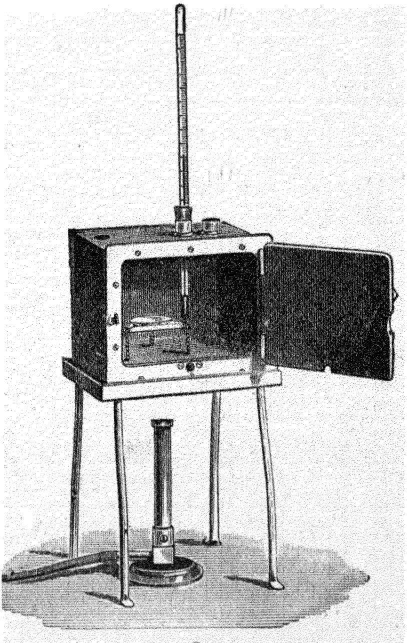


CONSTANT WATER-FEED.

The air-oven (fig. 59) may be employed instead of the steam-oven, the temperature of the interior being suitably adjusted by noting the thermometer and properly regulating the flame.

A precipitate may be more rapidly dried by first draining it in the funnel, and then removing the filter and spreading it upon a piece of wire-gauze which is supported on the ring of a retort-stand or upon a high tripod-stand. A small flame from a rose-burner is then placed beneath the gauze, at a sufficient distance to prevent any risk of charring the paper.

FIG. 59.



AIR-OVEN.

A precipitate may be partially dried by opening out the filter, which contains it, upon several dry filter-papers, and allowing these papers to absorb the water. This process may precede those which have been already mentioned.

REMOVING THE PRECIPITATE FROM THE FILTER.

99. Removal of Part of the Precipitate from the Filter.—A small quantity of a moist precipitate may be taken from the filter by dipping the end of a glass rod into it. If a watch-glass, or the interior of a test-tube, is then touched with the end of the rod, a portion of the precipitate is deposited upon the surface of the glass, and may be subjected to further test or examination.

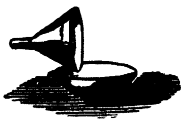
A larger quantity of the precipitate may be taken out of the filter by means of a glass spatula, made by flattening out the end of a glass rod which has been softened in the flame.

100. Removal of the Whole of the Precipitate from the Filter.—If the precipitate is to be removed from the filter as completely as possible, several methods are available. One or other of those described below under **a**, **b**, **c**, **d**, **e**, and **f**, must be chosen according to circumstances.

(a) **Washing down the Precipitate through the neck of the Funnel.**—A hole may be made in the bottom of the filter by means of a glass rod, which is pushed down through the neck of the funnel. The precipitate is then easily washed down into a vessel, placed beneath the funnel, by a fine stream of liquid from the wash bottle.

(b) **Washing out the Precipitate from the Rim of the Funnel.**—The funnel may be held with its neck horizontal, and with its rim just inside the edge of a porcelain dish (fig. 60). The precipitate is then washed out of the funnel by directing a fine stream of water from a wash-bottle against the inside of the filter.

FIG. 60.



WASHING THE PRECIPITATE OUT OF THE FUNNEL.

(c) **Removal of the Filter with the Precipitate from the Funnel, followed by rinsing off the Precipitate.**—The precipitate is allowed to remain in the filter in the funnel for some time, in order to permit as much water as possible to drain away. The filter is then carefully taken out of the funnel and is further dried if necessary, by laying it upon several folds of filter-paper. After the portions, which contain no precipitate, have been removed, the filter is spread out inside a porcelain dish.

The liquid, with which the precipitate is to be treated, is then poured into the dish; and this liquid is shaken round in the dish, and the filter is carefully rubbed with the rounded end of a glass rod, until the precipitate has been removed from the paper. With a little care this may be effected without tearing the paper. The filter-paper is then removed from the liquid by means of a glass rod.

(d) **Removal of the Filter with the Precipitate, followed by Scraping off the Precipitate.**—If it is undesirable to add a liquid to the precipitate upon the filter, the precipitate is allowed to drain for a short time, and is then further drained, if necessary, by removing the filter from the funnel and laying it upon several dry filter-papers.

The filter is then spread out upon a flat piece of glass, and the precipitate is carefully scraped off with a glass rod which is pressed flat upon the paper, or with a small glass spatula (99).

This method is usually the most imperfect, but is frequently the best for other reasons.

(e) **Removal of the Precipitate by Dissolving it in the Filter.**—If a precipitate is to be dissolved off the filter, the liquid, which is to be used as a solvent, is heated, and is then poured upon the precipitate. The solvent will run through the filter into a vessel placed below the funnel, and will take with it the precipitate in solution.

After the liquid has passed through the filter, it should be heated again and once more poured upon the precipitate if the latter is not entirely dissolved. The liquid is reheated and returned to the filter in this way as long as anything remains undissolved. If any portion of the precipitate still remains, it must be removed by using a fresh portion of the solvent.

EXPERIMENT 76.—Remove in this way the precipitate of barium carbonate (Exp. 74) from the filter, by means of hot dilute hydrochloric acid.

(f) **Removal of the Precipitate by Rinsing it through the Filter with the Solvent.**—A precipitate may also be removed from the filter, by means of the liquid with which it is to be treated or dissolved, in the following manner.

The funnel is supported over a suitable vessel. The liquid is poured in and is quickly stirred up with the precipitate by means of a thin glass rod. The bottom of the filter is then pushed out through the neck of the funnel by the glass rod. The liquid will now flow through the neck of the funnel, carrying the precipitate with it.

If some of the precipitate remains on the filter, the liquid must be poured again through the funnel, and by repeating this process all the precipitate may be removed.

USE OF TEST-PAPERS.

101. Litmus and Turmeric Test-papers.—The two vegetable colouring substances, *litmus* and *turmeric*, undergo marked changes of colour when they are acted upon by certain substances. These changes serve to indicate whether a liquid or gas is *acid*, or *alkaline*: if no change occurs, it may be inferred to be *neutral*.

The analyst is usually provided with slips of paper which have been stained with solutions of these colouring substances. These slips of paper are known as *test-papers*. The different papers are easily distinguished by their colours. The following experiments will explain their use.

102. Acid, Alkaline, or Neutral Reaction in a liquid is ascertained by the behaviour of the liquid with these test-papers.

EXPERIMENT 77.—Dip a clean dry glass rod successively into dilute hydrochloric acid, into ammonium hydrate solution, and into barium chloride solution. After each immersion of the rod, draw its end, wetted with the solution, successively across blue litmus-paper, red litmus-paper, and yellow turmeric-paper. The following results will be obtained.

Hydrochloric acid will redden blue litmus, but will not alter the colour of red litmus or of turmeric; it possesses an *acid reaction.*

Ammonium hydrate solution will not affect the colour of blue litmus, but will turn red litmus blue and turmeric brown; it shows *alkaline reaction.*

Barium chloride solution will not affect the colour of any one of the test-papers: it is *neutral.*

Each of these liquids is typical of a large class of substances, in so far as its behaviour with these test-papers is concerned.

103. Neutralisation: Addition of Acid or of Alkali in Excess.—It is occasionally necessary to exactly neutralise an acid liquid by means of an alkaline liquid or *vice versâ.* The acid or alkali must also often be added in excess. The following experiment explains how these processes are effected.

EXPERIMENT 78.—Pour some dilute hydrochloric acid into a porcelain dish. Add solution of ammonium hydrate drop by drop, constantly stirring the liquid with a glass rod and touching the wet end of the rod against a piece of blue litmus-paper.

By proceeding cautiously, a point may be reached at which the liquid does not affect the colour of either blue or red litmus-paper; the acid is then said to have been *neutralised* by the alkaline liquid. The latter part of the process of neutralising the acid liquid should be completed by the addition of the alkaline solution in a very dilute condition, since the addition of an excess of the alkaline solution is thus more readily avoided.

Add another drop of ammonium hydrate: the liquid will now have acquired an alkaline reaction: the alkaline solution is accordingly said to have been added *in excess.*

It will be easily understood from the above description how an alkaline solution may be neutralised by the addition of an acid, and how it may be *acidified* by the addition of an *acid in excess.*

104. General Remarks on Test-papers.—In selecting between red litmus and turmeric test-papers for the detection of alkaline reaction, it should be remembered that turmeric-paper is usually more sensitive than red litmus-paper, since red litmus-paper frequently contains an excess of acid.

A turbid liquid will often form a deposit when it is placed upon the test-paper, and this may conceal the colour of the paper. The colour will, however, usually be visible on the back of the paper if it is unglazed. If the paper is glazed, the colour will be easily seen after the deposit has been washed off by a stream of distilled water from the wash-bottle.

Test-papers must always be kept in a stoppered bottle, in order to guard them against undergoing change by the action of gases contained in the laboratory air. The bottle should be shielded from light as much as possible, since these vegetable colours fade under the action of strong daylight: this is especially the case with turmeric paper.

SIMPLE IGNITION.

105. Solid Substances are Heated strongly, or Ignited, in order to study the effect of heat upon them, since by this means their composition or nature is frequently revealed more or less completely. Ignition also serves to remove volatile from non-volatile substances.

It is a necessary condition of the process of *simple ignition* that the substance, which is being heated, shall suffer change only by heat. It must not undergo chemical alteration by contact either with the flame, or with the substance of the vessel or support, or by admixture with chemical reagents.

EXPERIMENT 79.—Heat some white **zinc oxide** in a small ignition-tube (8). The hot substance will be *lemon-yellow*, but the colour will change to *white* again as the oxide cools. These changes of colour are characteristic of zinc oxide.

In the early stage of the heating, *drops of water* will probably condense in the upper part of the tube: this proves that moisture was present in the oxide. Ignition in a tube is often employed to detect the presence of water in a substance.

EXPERIMENT 80.—Heat a little **mercuric oxide** in a small test-tube or ignition-tube. After a short time minute shining drops of the metal mercury will be seen in the upper part of the tube. If a splinter of wood with a spark at its end is now held in the tube, it will burst into flame. The behaviour of this red powder, when it is ignited, shows that it contains the elements mercury and oxygen (25).

EXPERIMENT 81.—Heat a few particles of **charcoal powder** on platinum foil, by directing the tip of the blowpipe-flame against the under surface of the foil. The charcoal will slowly burn away. This behaviour shows that the black substance consists of carbon.

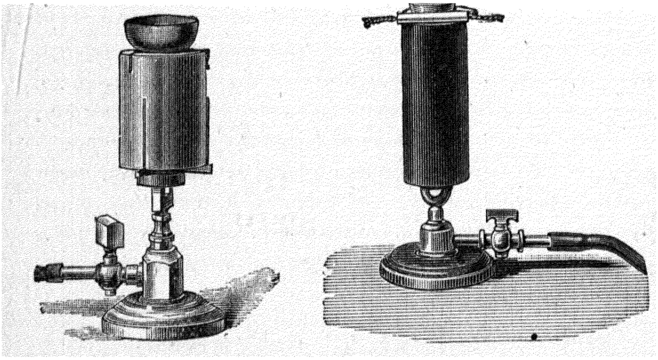
EXPERIMENT 82.—Heat a small piece of **ammonium**

chloride in an ignition-tube. The ammonium chloride will pass off as vapour or *volatilise*, and the vapour will cool in the upper part of the tube forming a coating or *sublimate*. This proves that the salt is volatile.

EXPERIMENT 83.—Heat in the same way a small piece of **potassium chloride.** This will melt, but will not pass off as vapour. It will become solid again on cooling. Potassium chloride is only very slightly volatile.

Hence if a mixture of ammonium chloride and potassium chloride is heated on platinum foil, the potassium chloride alone will be left in the residue, since the ammonium chloride will be removed as vapour.

FIG. 61. FIG. 62.



IGNITION OVER THE WALLACE-BURNER.

If the quantity of the substance to be ignited is large, it is usually heated in a porcelain or platinum vessel over the Bunsen- or Fletcher-flame (fig. 80, 1076).

Small quantities of substance may be conveniently ignited in little porcelain or platinum capsules over a Wallace-Argand burner from which the gauze top has been removed (figs. 61, 62).

The luminous flame must not be allowed to touch the capsule.

TREATMENT WITH FUSED REAGENTS.

106. **A Substance is often Heated with certain Solid Reagents** until they fuse, in order to cause chemical changes to occur between the substance and the reagents.

EXPERIMENT 84.—Mix a little **barium sulphate**, which is insoluble in water and in acids, with sodium and potassium carbonates, and heat the mixture on platinum foil until the alkaline carbonates fuse. Maintain the salts in a fused condition for several minutes. The following change will have taken place:—

$$BaSO_4 + Na_2CO_3 = BaCO_3 + Na_2SO_4.$$

Now treat the cool product of the fusion with water. Sodium sulphate will be dissolved. Separate the residual barium carbonate by filtration, and wash it. Then treat it with dilute HCl: the insoluble carbonate will dissolve as chloride. The original barium sulphate is thus obtained *in solution* as chloride.

Substances which cannot be dissolved in water or in acids may frequently be obtained in solution in a similar way to that just described. The mixture of alkaline carbonates, which is used for the purpose, is generally known as "fusion mixture."

Sometimes the treatment with fused reagents yields a characteristic result, which serves to detect the presence of a substance.

EXPERIMENT 85.—Place a little powdered **sodium carbonate** and **potassium nitrate** on a piece of platinum-foil, and then add a minute quantity of **manganese dioxide**. Hold the foil with crucible-tongs in the top of the Bunsen-flame, or heat the *under surface* of the foil with the point of the blowpipe-flame. The mass will soon melt or fuse. Keep the substances in a melted state for a short time, and th allow the mixture to cool.

A *bluish-green* mass will remain upon the foil, the colour of which is due to the sodium manganate which has been formed by the fusion. This colour is produced only when manganese is present, and the above process therefore serves as a test for that metal.

In the above experiments (79–85) the gases of the flame are not allowed to take any part in the change which is described. In other cases of ignition, which immediately follow, the flame gases aid in producing the reaction.

Flame Reactions with Reagents.

107. **The Gases of the Blowpipe-flame** (6) **or of the Bunsen-flame may play the part of Reagents** in affecting the composition of the substance which is being heated. In order to understand the nature of these changes the structure of the flame must be carefully observed.

In a well-formed blowpipe-flame or Bunsen-flame two parts may be distinguished: the *inner-flame*, which is blue and pointed; and around and beyond this an almost colourless flame, which is commonly known as the *outer-flame*.

The Inner-flame is often called the *reducing or the deoxidising flame*, because it separates or reduces metals from their oxides and from many of their compounds, and in general deoxidises substances. This deoxidising power is due to the action of the hot but incompletely burnt combustible matter, which is present in this part of the flame.

The inner flame is obtained of large size by placing the tip of the blowpipe nozzle only in the very edge of the flame, which has been made somewhat luminous by partially closing the air-holes of the Bunsen-burner.

The Outer-flame is also known as the *oxidising-flame*, ecause it changes metals into their oxides, and oxidises ny other substances, by supplying them with oxygen at a temperature.

The size of the outer-flame is increased by pushing the nozzle of the blowpipe into the interior of a Bunsen-flame, which has been rendered partially luminous by properly adjusting the air-supply.

In the following processes the results obtained will usually vary when the substance is heated in the inner-flame or in the outer-flame.

108. The Borax-bead—Fusion with Borax.—When certain metals and their compounds are fused with borax, they are converted into coloured borates. The colour of each of these borates is characteristic, either in itself or in the changes which it undergoes in the outer and inner flames.

The method of making the borax-head is explained in Exp. 86. Its use for testing is illustrated by Exp. 87.

EXPERIMENT 86.—Place a little powdered borax on a watch-glass. Moisten the loop of a piece of mounted platinum-wire (9), and dip it into the borax. Then hold the loop with the adhering powder in the outer blowpipe-flame until the borax melts.

If the bead thus formed does not fill the loop, dip the melted bead again into the borax; more powder will adhere and may be melted in the flame. In this way powder is to be constantly added, until a drop or bead is obtained which completely fills the loop. This bead must be perfectly colourless and transparent when it is cold.

If the melted bead easily drops from the wire, either the loop or the bead is too large. A loop which is somewhat less than an eighth of an inch across answers well; it may be made by rolling the tip of the wire round a piece of stout wire. If the bead is too large, remove a portion of it by giving the wire a slight jerk while the borax is in a fused condition.

If the bead thus formed shows any colour, it is jerked off while it is in a fused condition, and another bead is made from fresh borax. This process is repeated, if necessary, until a bead is obtained, which is colourless after it has been heated in the outer blowpipe-flame.

Experiment 87.—Moisten the clear cold borax-bead, and dip it into some finely-powdered manganese dioxine; *a few minute particles* only are to be thus attached to the bead. Then fuse this substance into the bead, by heating it for a short time in the tip of the *outer* blowpipe-flame. Examine the colour of the bead at once by looking through it at a piece of white paper, or at a window-light or bright flame. It will be *purple* or *amethyst-red* while hot, and the colour will not alter when the bead has cooled.

Now heat the bead again for some time in the tip of the *inner* blowpipe-flame. Its colour will vanish. The colour will, however, reappear when the bead is heated at the tip of the outer blowpipe flame. These changes of colour in the borax bead characterise the metal manganese.

General Remarks on the Borax-bead.—A beginner often obtains an opaque bead in the above experiment. This is caused by the use of too much manganese oxide. If this should happen, the bead should be fused again, and a portion of it should be shaken off and replaced by fresh borax.

When the test with the borax-bead is finished, the bead is detached, while it is still fused, by giving the wire a sudden jerk. The bead may also be removed, after it has cooled, by crushing it on a hard flat surface by a sharp blow with the pestle.

Two mounted platinum-wires should be kept, and their loops should always be immersed in dilute hydrochloric acid when the wires are not in use. They will then only require to be washed with water before they are employed for the above test.

109. The Microcosmic-bead.—Certain substances give more decisive colorations to a fused bead of microcosmic salt ($NaNH_4HPO_4.4H_2O$) in the platinum-wire loop than to a bead of borax prepared as has been described in the preceding paragraph. The platinum-wire loop for this test must be made smaller than for borax, else the bead drops out of the loop.

110. Ignition on Charcoal in the Blowpipe-flame.—Wood charcoal is often employed as a support for substances which are to be strongly heated in the blowpipe-flame. It possesses the advantage of being cheap and infusible, and of being a very poor conductor of heat.

Charcoal is often, however, selected for this use on account of the reducing or deoxidising power which it can exert at a high temperature. This renders it entirely unsuitable for oxidising processes such as that described in Exp. 85 (**106**).

Ignition on charcoal, therefore, serves chiefly to detect the presence of the heavier metals in their compounds. These are smelted out by the reducing action of the inner blowpipe-flame, aided by that of the red-hot charcoal. The assistance of fused reagents is, however, often necessary or at least advantageous.

The vapours of some metals, which have been thus smelted out, burn as they leave the charcoal and the metallic oxides thus formed are deposited as *incrustations*. The white ash, which is left by combustion of the charcoal, must not be mistaken for such an incrustation.

EXPERIMENT 88.—Choose a piece of wood-charcoal which is free from large cracks, and which does not crackle or spit when it is heated in the blowpipe-flame. Scoop out a small hollow near one end of it, with a knife or with a charcoal-borer.

Place in this cavity a little powdered lead acetate and sodium carbonate, and mix the powders together in the cavity with the point of a penknife.

Heat this mixture in the inner blowpipe-flame, holding the charcoal in such a way that the flame plays over its surface (fig. 63).

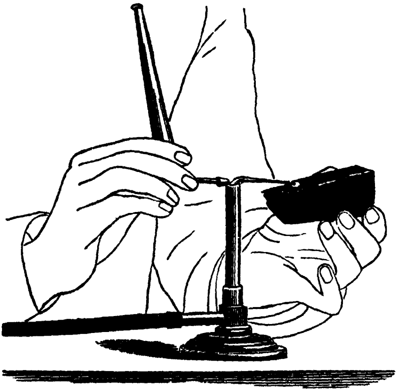
Bright globules of lead will soon be visible in the cavity; and when the charcoal is removed from the flame, the surrounding surface will be found to be coloured with a *yellow incrustation.*

Take one of the little globules of metal from the charcoal with the point of a penknife, and strike it smartly with the pestle on the bottom of the inverted mortar. It will flatten out into a cake, showing that the metal is *malleable* and not *brittle*.

Detach another metallic globule, cleanse it from charcoal by rolling it between the moistened finger-tips, transfix it on the point of the penknife blade, and rub it on some paper. It will be found to mark paper as black-lead does.

Lead compounds are characteristic by giving a yellow incrustation, and malleable globules which are soft enough to mark paper, when they are subjected to this test on charcoal. The test therefore serves to detect the presence of lead in its compounds.

FIG. 63.



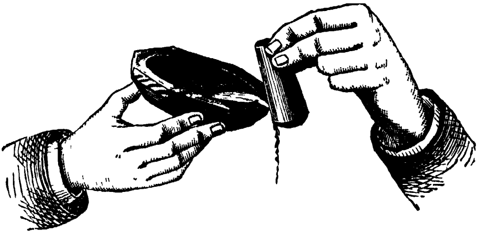
IGNITION ON CHARCOAL.

111. Washing and Levigation of the Residue left on the Charcoal.—Metallic globules, which have been obtained according to the directions given in the preceding experiment, are usually visible, at least with the aid of a lens. They are also easily detached from the charcoal.

But fine metallic particles or scales can often only be detached and separated, after the fused residue has cooled and has been detached from the charcoal with the point of a penknife, and has then been washed by levigation.

With this object, the residue is crushed in a mortar, or in a porcelain dish, with a little water. The water is then quickly decanted down the wet pestle, which is pressed against the edge of the vessel (fig. 64). The light suspended particles of charcoal, together with soluble substances, are thus poured off; while the heavier and insoluble metallic particles are left in the vessel. By repeating this process of *levigation*, the metal may be obtained free from soluble substances and from charcoal; it may then be further examined by a lens, or may be tested with a magnet or by other means.

FIG. 64.



WASHING BY LEVIGATION.

It may be noted that the addition of a piece of potassium cyanide to the mixture, before it is heated on the charcoal, will usually much facilitate the process of smelting out the metal, and will assist in bringing it into the globular condition.

EXPERIMENT 89.—Fuse some sodium carbonate with a little copper sulphate on charcoal in the inner blowpipe-flame, and levigate the residue as is directed above. Little *red scales* of copper will be obtained.

EXPERIMENT 90.—Treat in the same way a mixture of ferrous sulphate and sodium carbonate. A *grey powder*, consisting of metallic iron, will remain. When this powder is touched under water with the point of a magnetised penknife, it will attach itself in the form of a tuft.

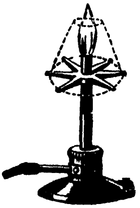
FLAME-COLORATIONS.

112. Characteristic Colours are imparted to a Non-luminous Flame by the vapours which many substances emit at a high temperature.

The blowpipe-flame may be employed to obtain these colorations. A Bunsen-flame also answers the purpose well, but on account of the lower temperature of this flame, many colorations are not obtained with promptness or intensity.

When the Bunsen-flame is used for this purpose, it may be partly surrounded with a conical chimney, which steadies the flame considerably (figs. 65, 66, 67). The supply of air, entering by the air-holes, should be so regulated that a small luminous point appears near the top of the flame. The platinum wire is held either in this luminous point or just below it.

FIG. 65.



BUNSEN-BURNER WITH CHIMNEY.

EXPERIMENT 91.—Heat a platinum wire loop, which is not more than an eighth of an inch in diameter, in the inner blowpipe-flame. If the wire is clean, it will become red hot without colouring the flame.

If the wire colours the flame, it must be boiled with hydrochloric acid in a test-tube and then rinsed well with water. Or it may be wetted with strong hydrochloric acid, and then heated strongly in the blowpipe-flame until it no longer colours the flame. If the loop cannot be cleansed in this way, the end of the wire must be cut off, and a fresh loop must be formed.

Now dip the wire-loop into a little sodium chloride solution on a watch-glass. When the loop is removed, it must remain filled by a drop of the solution. Then hold the loop at the tip of the inner blowpipe flame. The outer flame will be coloured *bright yellow.*

Repeat the experiment, placing the loop containing the solution in the outer part of the Bunsen flame, about halfway between its base and top. The same yellow tint will be seen above the loop.

This yellow coloration is given to the flame only by sodium and by its volatile compounds. On looking at it through the indigo-prism, no yellow coloration will be visible.

EXPERIMENT 92.—Remove all sodium chloride from the platinum wire loop by heating it strongly in the tip of the blowpipe flame as long as it imparts any colour to the flame. Then dip the wire-loop into some solution of *pure* potassium nitrate. This will impart to the flame a *pale lilac* colour, which is characteristic of potassium and its compounds. When this coloration is viewed through the indigo prism it will appear *crimson*.

EXPERIMENT 93.—Now dip the wire loop into a mixture of a few drops of the sodium chloride solution with a few drops of the potassium nitrate solution, which has been made on a watch glass. When the loop is held in the flame the sodium coloration only will be seen. But if this coloration is then viewed through the indigo prism, the crimson potassium coloration will be seen distinctly. By means of the indigo-prism the potassium coloration is therefore discoverable, even when it is concealed by that of sodium.

THE SPECTROSCOPE.

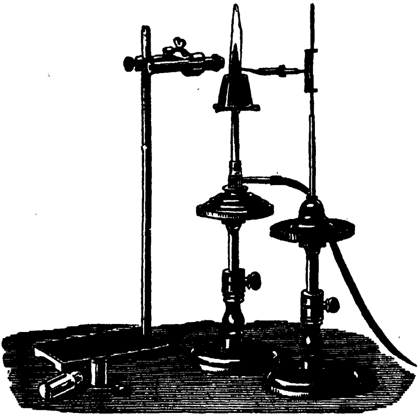
113. The Spectra furnished by the Spectroscope afford the most certain means of detecting substances by means of their flame colorations, more especially when the colorations due to several substances are mingled in the flame.

When the coloured light from the flame coloration passes through the spectroscope it is separated into its constituent colours by traversing a glass prism. The coloured lines, which are seen as the result of this separation, are termed the *spectrum* of the substance. By their colours and relative positions they serve to identify the substance with certainty.

This is equally true whether the flame-coloration is produced by one substance only, or by several; since the constituent colours are in any case seen without interference one with another.

Two forms of spectroscope are in common use for chemical analysis. *The Direct-vision Spectroscope*, which is shown ready for use in fig. 66, is recommended by its compactness and its portability. In its smallest form it can be carried in the waistcoat pocket. *The Bunsen or Table Spectroscope*, shown in fig. 67, is to be preferred for general use as a stationary piece of apparatus in the laboratory.

FIG. 66.



THE DIRECT-VISION SPECTROSCOPE.

The light enters each of these instruments through a slit, the width of which can be adjusted to suit strong or feeble colorations. The rays of light, after passing the slit, are rendered less divergent by traversing a lens, and then pass through one or more prisms. The rays are finally focussed by means of a sliding eye-piece, so as to give a sharp image of the slit.

114. The Direct-vision Spectroscope is arranged for use by supporting it in a clamp in front of the flame (fig. 66).

The slit is then slightly opened, and the eye-piece is adjusted so as to sharply focus the vertical yellow line, which is due to the sodium compounds present in the atmospheric dust.

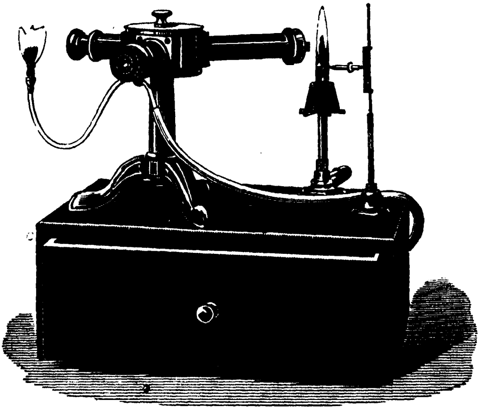
Instead of supporting the instrument by a clamp, it may be held in the hand and directed on the flame. It is then most readily focussed, before it is directed on the flame, by rendering sharp the dark Fraunhofer lines, which are seen on looking through a window at ordinary daylight.

Proceed to Exp. 94.

115. The Bunsen or Table Spectroscope (fig. 67) has usually three brass tubes. One of these carries the adjustable slit, another the eye-piece, and the third a transparent photographic scale.

The instrument is brought into adjustment by placing a Bunsen-burner with closed air-holes in front of the slit.

FIG. 67.



THE TABLE SPECTROSCOPE.

On looking through the eye-piece at this luminous flame, a *continuous spectrum* of unbroken colours will be seen. The upper and lower edges of this spectrum are sharply focussed by moving the eye-piece, and the two tubes are arranged at such an angle as to give the longest complete spectrum possible. The tubes must either be permanently clamped in this position, or they must be so marked that they can at any time be brought readily into the same position with certainty.

Air is then admitted into the Bunsen-flame by opening the holes of the burner, and the yellow sodium line, which is always visible, is arranged in a vertical position by rotating the slit-tube, and is then sharply focussed. The bright flame is now lighted opposite the end of the scale-tube; the scale is focussed; and the scale-tube is shifted until the yellow sodium-line stands at a particular graduation, the fiftieth being selected for the special chart in the figure. This yellow line will usually be seen in all the following experiments, since it is derived from the atmospheric dust, which enters the flame: it may be produced, if necessary, by introducing a platinum wire, moistened with sodium chloride solution, into the flame.

Experiment 94.—When the spectroscope has been brought into adjustment, the student should proceed to introduce a mounted platinum wire (9) into the front of the Bunsen-flame, at a level just below the slit of the spectroscope. The platinum wire loop should be moistened successively with solution of sodium chloride, of potassium chloride, of barium chloride, of strontium nitrate and of calcium chloride.

The wire may be conveniently supported in the flame by slipping its glass handle upon a horizontal wire arm, the height of which is adjustable on a stand such as is represented in figs. 66, 67.

Coloured vertical lines will be seen when these flame-colorations are observed through the spectroscope. These lines will always be the same in colour and position for the same flame-coloration. They will vary, however, in these respects with the colorations of different substances. A particular *line-spectrum* is therefore characteristic of each of the vapours which give rise to the above flame-colorations.

116. The Spectrum-Chart.—The pocket-spectroscope is not usually furnished with a graduated scale for measuring the position of the spectrum-lines. In the absence of a scale the relative positions of the lines of any particular spectrum may, however, be jotted down or remembered with approximate accuracy. Since the yellow sodium line is never absent, it will furnish a useful landmark in the spectrum.

The scale of the larger spectroscope, however, enables the position of the spectrum-lines to be noted down on a chart, and they are thus easily identified if the instrument is always brought to the same adjustment. The instrument must always be brought to precisely the same adjustment as it possessed when the lines were mapped down in the spectrum-chart, if the chart is to be of any use.

The spectrum-lines will vary in position in different instruments, owing to variation in adjustment, and to difference in the material and angles of the prisms. Hence it is necessary to make a separate chart for each spectroscope.

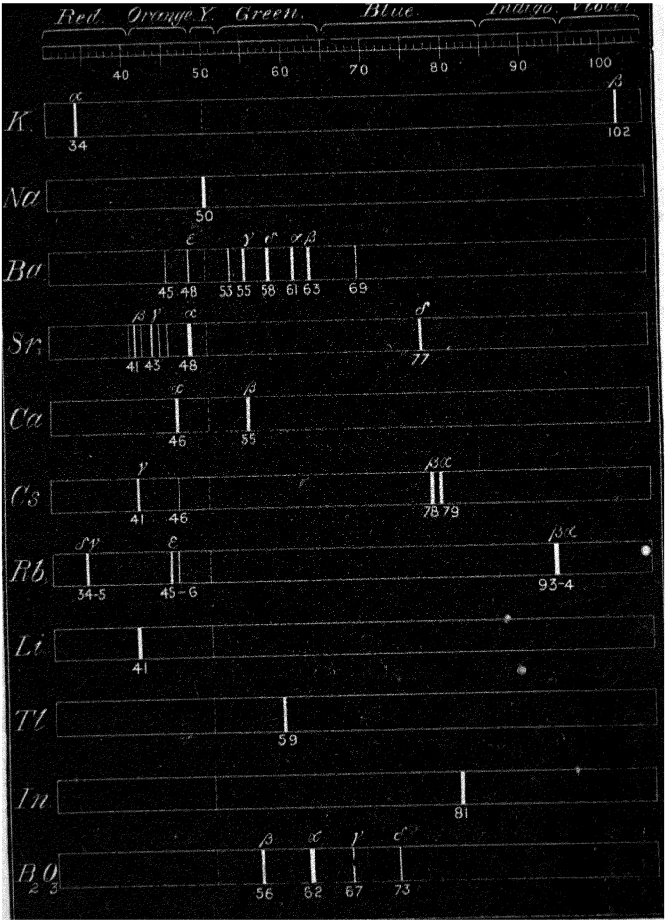
The accompanying spectrum-chart (fig. 68) indicates a convenient method of mapping the lines, but the position of the lines on the scale only applies to the spectroscope from which the chart was made.

In this chart the relative intensity of the spectrum-lines may be inferred from their varying thickness, while the relative intensity of the lines in each spectrum is indicated by the Greek letters applied in the order of decreasing intensity. The colour of each line is shown by the name of the colour which is placed at the top of the chart, over the region in which the line occurs. The position of each line is indicated by placing beneath it the number of the scale-graduation at which it stands. The position of the yellow sodium-line is marked in each spectrum by a dotted line: this line is usually seen in every test which is made, for reasons which have been already stated, and it therefore serves as a landmark.

A chart of this kind must be at hand for reference when the spectroscope is used for analytical work. The spectrum-lines may be marked in with pen or pencil on a white ground, but they are more striking if coloured. The chart may conveniently be suspended near the instrument.

The spectrum of an unknown substance may be identified, even when the instrument has not been carefully adjusted, and in the absence of the chart. For this purpose the position of the lines is noted on the scale and the flame-coloration

FIG. 68.



THE SPECTRUM-CHART.

of a known substance, which is believed to correspond to the spectrum which is under observation, is then thrown into the spectroscope. If the spectrum-lines of the unknown substance are identical in colour and in position with those given by the known coloration, the two spectra must be due to the same substance, and the unknown substance is therefore identified.

EXPERIMENT 95.—The student should carefully observe and map the spectra of several of the metals, which are mentioned in the spectrum-chart (fig. 68). He should then proceed to examine some of the mixed spectra, which are obtained from solutions containing the salts of two or more of these metals. The ease with which many metals can be found by means of the spectroscope, even in the presence of one another, will thus become evident.

The Delicacy of this Method of detecting certain metals is extreme. Thus it is only in air which has remained undisturbed for a long time and is almost absolutely free from dust particles, that the sodium spectrum is not obtained from a Bunsen-flame. By using extremely dilute solutions of metallic salts, the delicacy of the method has been proved in the case of other metals. Bunsen states that one three-thousand-millionth of a grain of sodium can be detected by the spectroscope.

In the more Powerful Modern Spectroscopes several prisms are used, and the lines of the spectra become more widely separated. Their relative positions may then be noted with great precision by moving the observing-tube until each line in succession is brought into the middle point of the cross-wires in the eye-piece. The position of the eye-piece is then read off on the graduated edge of the circular table on which the prisms stand, and registers the position of each line. Instruments of great power and precision are however not requisite for ordinary analytical work, and the simple form of table spectroscope which has been already described will be found to be sufficient for the purpose.

117. Use of the Spectroscope in Chemical Analysis.—

The spectroscope is of special use in chemical analysis for the following purposes.

1. For readily detecting metals when their flame-colora-

tions are concealed by that of sodium; and generally for detecting metals, when their colorations are mixed in the flame.

2. For distinguishing similar colorations from one another. Thus the red coloration of strontium is easily distinguished from that of calcium, by the strongest red line in each of the spectra being at a different distance from the sodium line; these very similar flame-colorations are further distinguished by the appearance of a characteristic blue line in the spectrum of strontium, and by a characteristic green line in the spectrum of calcium.

3. For detecting minute traces of many metals, which could scarcely be discovered by other means. The metals lithium, indium, thallium, rubidium, and cæsium were discovered by their spectra; and as these metals usually occur in minute proportion only, they are most conveniently detected by the spectroscope.

It is by no means a matter of indifference which compound of a metal is selected for obtaining the flame-coloration or spectrum of the metal. The most volatile salts are generally the chloride, the nitrate, and the chlorate; these accordingly give the most vivid but the least durable spectra. The coloration is increasingly feeble with the sulphate, the carbonate, and the phosphate; and the silicate does not usually give any coloration at all.

The coloration becomes more intense when the finely powdered substance is moistened with strong hydrochloric acid. But in the case of a silicate it is usually necessary to moisten the powder with hydrofluoric acid, or to mix it with pure ammonium chloride and calcium carbonate, before it is introduced into the flame.

Some very volatile substances give only a momentary flash of colour in the flame; while others, which are far less volatile, colour the flame only at the highest temperature obtainable. Hence careful observation of the flame coloration through the spectroscope is necessary, *from the moment* that the substance is introduced into the flame, until the wire-loop has been heated to whiteness for some time.

The width of the slit of the spectroscope should be regulated according to the intensity of the lines to be looked for. A bright line can be observed with a narrow slit, and its position is thus noted with great precision. The slit must, however, be opened much more widely to see a faint line, such as Kβ (fig. 68). Great breadth of the line is objectionable, since it prevents its position on the scale from being accurately noted, and often causes the edges of neighbouring lines to overlap.

The background of the flame should be absolutely black, in order to prevent admission of stray light into the slit. A piece of black velvet hung behind the flame gives a good background.

SECTION IV.

ANALYTICAL REACTIONS.

PART I.—INTRODUCTION TO THE ANALYTICAL REACTIONS.

Some paragraphs in this Section are marked with an asterisk (*) These may be omitted, if the analysis of simple salts only is to be learnt. These paragraphs will, however, require the careful attention of the general analyst.

Reactions printed in small type are of minor importance only, and may be passed over, or simply read through, if time is limited.

INTRODUCTORY REMARKS.

126. Object and Method of Chemical Analysis.—The study of chemistry has revealed the fact that every substance consists either of one kind of matter only, or of two or more different kinds. Those substances which contain only one kind of matter are called *Chemical Elements.* Their number at present amounts to about seventy. A list, which comprises most of them, is given at the end of the book (**1173**).

These elements do not usually occur singly. Two or more of them are commonly found together either in a state of mere *Mixture,* or united in a much more intimate manner in a *Chemical Compound.*

It is the object of analytical chemistry to discover what element, elements, or sometimes what groups of elements or ***Radicles,*** any substance of unknown composition contains.

But an analysis may also be undertaken in order to ascertain whether any particular element or group of elements is present in a given substance.

An analyst should be acquainted with the properties of each element and of the compounds which it forms with other elements. Efficiency in analysis therefore depends in a large measure upon the knowledge of what is usually termed *Theoretical Chemistry*, but may be more appropriately styled *Descriptive Chemistry.*

For ordinary analytical purposes, however, certain marked properties or changes of each element, or group of elements, are selected; and by these properties, called *Tests* or *Reactions*, its presence is detected.

The tests or reactions which are most frequently selected depend upon the behaviour of a substance when it is heated under various conditions (105–117), and upon its behaviour when it is mixed with certain liquid or solid substances (90).

Such substances, when they are added for analytical purposes, are called *Reagents.* Some reagents serve for detecting one element or compound only, and are therefore called *Special Reagents;* others separate or detect a group of elements or compounds, and are called *Group Reagents* or *General Reagents.*

127. Analytical Groups.—The metals are separated, by their behaviour with certain general reagents, into five principal Groups. Two of these Groups are further subdivided into two Subgroups.

Each Group receives a distinctive name, which is derived either from its place in the system, or from some conspicuous or important member of the Group, or from the general reagent which is used to precipitate the Group. Thus we speak indifferently of the Fourth Group, of the Barium Group, or of the Ammonium Carbonate Group.

It is perhaps well to avoid referring to a Group by its number, since different analysts number the groups differently, and the number is therefore apt not to denote the Group with certainty.

At the beginning of the Fifth Section of this book (931) the Analytical Groups of the metals are tabulated; and the distinctive number, name, and reagent, which are characteristic of each Group, are placed at the head of its column. The names and symbols of the metals contained in the Group are arranged vertically beneath this heading.

128. Analytical Classification.—The chemical elements are frequently divided into two classes, *Metals* and *Non-metals*. These classes, with the following modifications, form convenient divisions for analytical purposes:—

I. With the metals must be classed the hypothetical metallic-radicle *Ammonium*, and the element *Hydrogen*.

II. The non-metals, either singly, or in groups formed by their combination *inter se* or less frequently with a metal, unite with hydrogen forming acids, and with metals forming *salts*. The acids may be looked upon as the hydrogen salts of these non-metals or of these radicles.

Thus Cl, SO_4, PO_4, MnO_4, form the hydrogen salts or acids, HCl, H_2SO_4, H_3PO_4, $HMnO_4$; and the metallic salts, KCl, $MgSO_4$, Na_3PO_4, $KMnO_4$. *Metal-hydrogen salts* or *Acid salts*, such as $NaHSO_4$, Na_2HPO_4, are also known.

The general name *Acid-radicle* is conveniently applied to Cl, SO_4, PO_4, MnO_4, and to similar elements or radicles. Some of these acid-radicles are elements, but most of them are unisolated *Compound-radicles*. The property which they possess in common is that of forming acids when they are combined with hydrogen, and of forming salts when they are combined with a metal.

For analytical purposes, then, substances may be separated into the groups of *Metals* and *Acid-radicles*; although certain elementary and compound substances cannot be included in this generally useful classification; and certain non-metallic or metalloidal substances, as well as certain acid-radicles, are for analytical purposes conveniently classed with the metals.

THE ANALYTICAL REACTIONS.

129. Trying the Analytical Reactions.—In order to become fully acquainted with the behaviour of different substances with *Reagents*, and thus to be able to detect these substances by their *Reactions*, each of the reactions should be tried with the substance itself or with one of its compounds. The appearances which are presented are carefully observed and remembered, and the substance may then be easily recognised in a body of unknown composition by means of these reactions or tests.

The addition of a reagent to a substance causes some important and visible change to occur. This may be simply a change of colour, but it very usually consists in the formation of a precipitate. The colour, the appearance, or the behaviour of this precipitate with other reagents, is characteristic of a particular element or compound-radicle.

As soon as a reaction has been satisfactorily performed, it should be briefly described in the note-book in a neat and concise manner. The nature of the chemical change which has occurred should then be explained by means of a chemical equation.

130. Chemical Notation and Chemical Equations.—The entry in the note-book of the chemical change, which occurs in an analytical reaction, involves the correct use of chemical symbols and formulæ, and a right conception of the meaning of a chemical equation For full information on these matters a treatise on theoretical chemistry should be consulted. The few hints which follow will, however, serve to guide and guard a student, who has not had much experience in the use of formulæ and of equations.

Chemical Notation.—Each element is represented by a *Symbol*, which consists of either one or two letters. Thus S stands for sulphur, Cl for chlorine. The symbol also represents *one Atom* of each element. When symbols are written one after another, they give the *Formula* of a compound substance. A correct formula shows what elements, and how many atoms of each of these elements, the compound molecule contains.

Thus KCl stands for one molecule of potassium chloride, a compound of one atom of potassium and one atom of chlorine. $PtCl_4$

stands for one molecule of platinic chloride, which consists of one atom of platinum combined with four atoms of chlorine; since the small figure, placed *below a symbol* to the right, denotes how many atoms of that element are present.

A number placed *before* a *formula* multiplies each symbol in that formula. Thus $2PtCl_4$ signifies two molecules of platinic chloride, containing altogether two atoms of platinum and eight atoms of chlorine. If two or more symbols are included in brackets, any number placed outside the brackets, and either *before* or *after* them, multiplies each symbol which is contained in the brackets.

Thus both $Sr(NO_3)_2$ and $Sr2(NO_3)$ are equal to SrN_2O_6. This may also be written $Sr2NO_3$, since the multiplication by 2 extends to each symbol which follows, until a break occurs in the formula. Thus $Sr2NO_3.4H_2O$ would denote $SrN_2O_6.H_8O_4$, if the multiplication were carried out.

In the Seventh Section of this book (1173) there will be found a list of the names of the Chemical Elements, with their symbols and atomic weights; also lists of the names and the corresponding formulæ of most of the compound substances which are referred to in the text (1160–1167).

Symbols and formulæ are always used in the text instead of names. The corresponding names are entered when the symbol or formula is used for the first time. These names may also be found by referring either to the lists mentioned in the preceding paragraph, or to the labels on the laboratory bottles.

Rules for Drawing out a Chemical Equation.—The student will understand that a chemical equation is a short statement of ascertained chemical facts, and that every equation which conforms to the general rules stated below is not necessarily true. For the special purposes for which equations are used in analytical reactions, however, the following rules will generally furnish true equations.

Write down the formulæ of the substances which are mixed together, with the sign of addition (+) between them. Then write the sign of equality (=), followed by the formula of the precipitate or product formed.

In most analytical reactions, a mutual exchange of certain elements or groups of elements, or a *double decomposition*, occurs between the two compounds which are mixed. A little consideration will therefore usually show how many molecules of these substances are required to yield the formula for the precipitate or principal product. It will also be seen whether any other substance is formed at the same time; and if it is formed, its formula may be easily deduced.

The number of atoms of any one element must always be the same on both sides of a correct equation.

Applying the above rules to the first test for potassium (141), we get :—

$$KCl + PtCl_4 = \overline{K_2PtCl_6}.$$

This statement is obtained by merely placing together the formulæ ot the substances mixed and of the precipitate formed.

It is evidently not correct, since there are two atoms of K on the right-hand side, and only one on the left; also six atoms of Cl are found on the right, and only five on the left. But this inequality is at once removed if we place 2 before the KCl, thus —

$$2KCl + PtCl_4 = \overline{K_2PtCl_6}.$$

Evidently if the above equation is correct, the precipitate is the only substance formed, since the number of atoms in the substances on the left-hand side is exactly equal to that in the substance on the right.

Take now the first test for Ba (170). Our tentative statement is :—

$$BaCl_2 + Am_2CO = \overline{BaCO_3} +$$

But here the left-hand side contains Am_2 and Cl_2 more than the right.

The equality is easily restored thus :—

$$BaCl + Am_2CO_3 = \overline{BaCO_3} + 2AmCl.$$

This equation states that when barium chloride ($BaCl_2$) and ammonium carbonate (Am_2CO_3) are mixed together, barium carbonate ($BaCO_3$) and ammonium chloride (AmCl) are obtained.

131. Abbreviations employed for Entry in the Note-book.—For convenience in hasty writing, it is not uncommon to employ abbreviated formulæ for the compound radicles. This is more especially the case with those radicles which are constantly met with, and with those which have a complex composition.

Only such abbreviations are employed in the text as save the use of brackets or of numbers. Thus Am_2 is a short rendering of $(NH_4)_2$, and Ho_6 of H_6O_6 or of $(HO)_6$.

The following abbreviations may be used :—

Am for (NH_4), the metallic-radicle ammonium.
$\overline{T}$ „ $(C_4H_4O_6)$, the acid-radicle of tartrates.
$\overline{A}$ „ $(C_2H_3O_2)$, the acid-radicle of acetates.
Cy „ (CN), the acid-radicle in cyanides.
$\overline{Cfy}'_4$ „ $(FeCy_6)$, in ferro- and ferricyanides.
Ho „ (HO), the radicle occurring in hydroxides.

The "hydrate" of a metal is preferred to "hydroxide" for reasons of brevity rather than for theoretical correctness.

The formulæ K_3FeCy_6, $FeCl_3$, As_2O_3 are frequently employed in place of the more complicated formulæ $K_6(FeCy_6)_2$, Fe_2Cl_6, and As_4O_6.

The following contracted words are also used:—

expt.	for	experiment.	*insol.*	for	insoluble.
pp.	„	precipitate	*sol^n.*	„	solution.
pp^n.	„	precipitation.	*cryst^{ne}*	„	crystalline.
pp^d.	„	precipitated.	*col^n.*	„	coloration.
sol.	„	soluble.			

132. Entry of Analytical Reactions.—An example is given below of the entry of the reactions in the Note-book. It will be seen that a systematic method is observed in writing out the equations. On the left-hand side of the equation the formula of the substance upon which the reaction is being tried is written first, then follows the formula of the reagent which is added. On the right-hand side of the equation, the formula of the precipitate or principal product is entered first, with a line drawn over it by way of distinction; then follows the formula of each substance which is formed at the same time.

In the subsequent reactions, the formulæ of the substance used, of the reagent added, and of the precipitate produced, will always be given. These data are generally sufficient to enable the student to form the equation. The equation will only be given in full when it is exceptionally difficult or complicated.

The following example of the brief entry of reactions in the Note-book has been selected from paragraphs **141-144**.

POTASSIUM (K).—Used KCl.

1. *$PtCl_4$*: stirred on watch-glass: yellow cryst^{ne} pp., sol in much water and in KHO, insol. in alcohol : $2KCl + PtCl_4 = \overline{K_2PtCl_6}$.
2. *$NaH\overline{T}$*: well shaken in a test-tube: white cryst^{ne} pp., sol. in water, in KHO, and in HCl : $KCl + NaH\overline{T} = \overline{KH\overline{T}} + NaCl$.

3. *Flame col^n.*: pale lilac, crimson through the indigo-prism ; a red and a faint violet line at the extreme ends of the spectrum.

4. *Heated in tube or on platinum foil* . solid KCl melted, but gave no fumes : hence it was not volatile.

133. Directions for Trying the Analytical Reactions.

1. A *solution* of the substance, the reactions of which are to be tried, must always be employed, unless it is specially stated that the *solid* is required. This liquid is often called *the original solution.* The solid substance is usually only required for blowpipe reactions.

A solution may be kept in readiness, or it can be easily made by warming the solid with water or with dilute acid (1163, 1164).

2. Pour into a test-tube or small beaker about twenty cubic centimetres of the solution of the substance : this constitutes the "original solution." If the solid substance is also required, take some of it upon a watch-glass and powder it finely.

3. Pour a little of the original solution into a perfectly clean test-tube and add the first-mentioned reagent to it. A few drops only of the reagent should be added at first ; more can be gradually added afterwards if necessary.

The habit of working with small quantities both of solutions and of reagents must be acquired from the first.

In trying each of the reactions a separate portion of the original solution is to be taken.

4. Before trying a reaction, the student should carefully read through *the whole of the description* of the results to be obtained. The experiment is then to be performed, literally following out the directions given.

It is a necessary condition of after success that each result should be obtained precisely as is stated in the text. The student must never on any account pass on without being satisfied that the statements of the book are true, and that he could at any time repeat the test successfully.

5. The water used for the tests and reactions must always be *distilled water.*

The acids are to be used in a dilute condition, unless it is stated that the strong acid is to be employed.

In all cases KHO and NaHO may be used indifferently the one for the other.

GENERAL RULES FOR WORKING.

134. The student should attend carefully to the following rules. Their observance will materially aid the progress and accuracy of his work.

1. Before commencing work, look through the reagent bottles belonging to the working bench (**1160**). Replace any which are absent, and see that the bottles are arranged in the order shown by the numbers on the labels. Then proceed to fill any which are empty, and to relabel any whose labels are imperfect or loose. Also filter any of the liquid reagents, which are not quite clear when they are shaken up.

2. Arrange the apparatus, which is required for use, on the back part of the bench, so as to leave the front of the bench free to work upon. This will lessen the risk of upsetting anything. (See Frontispiece.)

3. All dirty glass and porcelain, which is not in use, should then be carefully cleansed. This ought to be unnecessary, since apparatus should never be put away dirty.

4. When a reagent-bottle is to be used, take it in the right hand; remove the stopper by grasping it between two of the fingers, or between the fourth finger and palm of the left hand; hold the stopper in this way while the bottle is being used; then replace it immediately, and return the bottle to its place.

5. Pour a liquid reagent out of the side of the bottle

opposite to that which bears the label; and prevent the last drop of the liquid from running down the outside, by touching the lip of the bottle either with the stopper, or against the clean edge of the vessel.

6. Solid reagents should be taken out of the bottle on a clean knife-blade or horn spatula, or with a slip of clean card-board or paper.

7. *Each bottle must be restoppered and replaced on the shelf, in its proper place and with the label outwards, immediately after use; it must never be left standing on the working-bench.*

8. If a solution or precipitate has to be put by for some time before it undergoes further examination, it should always be labelled· a piece of gummed paper may be attached to it, bearing either an inscription, or a reference number which is explained in the note-book and which is sufficient to recall to mind what the solution or precipitate consists of. *It is well never to trust to memory in this matter.* It will be found to be unadvisable as a rule to leave a *precipitate* thus, since it is apt to dry or to undergo oxidation.

9. It should be remembered that if brass crucible-tongs are employed for holding hot evaporating basins containing acids, some of the brass is apt to be dissolved and thus introduced into the solution. A hot dish may be moved either by holding it with a cloth, or by lifting the stand upon which the dish is placed.

10. *Liquids only* should be poured down the sinks. All *solid* refuse, such as soiled filters, broken glass, and pieces of charcoal, should be thrown into a receptacle which is placed in an accessible position.

11. When a substance is being examined in order to find out its composition, the student should fully write down in the note-book each test or process, with the result obtained, *as soon as it is completed.* The analysis is thus written out gradually as it progresses. *On no account must the entry of the results be left until the completion of the analysis.*

This rule cannot be too strongly enforced, as neglect of it always causes uncertainty and confusion. The most convenient form of entry is that of the Analytical Tables.

12. *Cleanliness and neatness* in analytical work cannot be too carefully attended to. More confusion and error may be caused by using dirty test-tubes, beakers, dishes, and funnels, than would be thought possible by the beginner. Work should also be rendered as *systematic* as possible, in order to economise time and thought.

SECTION IV.—PART II.

ANALYTICAL REACTIONS FOR METALS.

In trying through the reactions for the Metals the usual order of the Analytical Groups has been reversed, because the most simple reactions occur in the last Groups and these are therefore better suited for a beginner.

Occasionally non-metallic or metalloidal elements are associated with Metal Groups for the purposes of analytical classification.

The reactions of the Rarer Metals belonging to each Group are entered in small type at the end of the Group.

Reactions printed in small type may be omitted, but they should be carefully read through.

A Table will be found in paragraph 1172, which contains the names and chemical formulæ of the more commonly occurring natural minerals, in which the different metals and acid-radicles are present.

GROUP V.—THE POTASSIUM GROUP.

140. This Group includes K, NH_4, Na, Mg, together with the rarer metals Li, Rb, Cs (164-168).

The members of this Group are not precipitated from their solutions by any Group-reagent.

POTASSIUM (K).—Use Potassium chloride, KCl.

141. Platinum chloride, $PtCl_4$, if it is added to some of the KCl solution on a watch-glass, and stirred gently but persistently with a glass rod, gives a yellow crystalline precipitate (K_2PtCl_6); this precipitate forms first along the lines where the rod has rubbed the glass.

Stir up the precipitate and pour off the liquid with the precipitate into three test-tubes.

To one of these portions add much distilled water and heat, the precipitate will dissolve. To another part add alcohol, the precipitate will not dissolve. Hence this precipitate is soluble in water and insoluble in alcohol.

By warming the third portion with solution of potassium hydrate (KHO), the precipitate is shown to be soluble in that liquid.

Remarks.—Since this precipitate is soluble in alkalies, a liquid, to which this test has to be applied, should always be made neutral or faintly acid before $PtCl_4$ is added. It is always well to acidify the liquid with dilute HCl, and to make sure by test-paper that it is slightly acid in reaction (102).

Further, since the precipitate is less soluble in alcohol than in water, the addition of alcohol often hastens the formation of the precipitate in dilute solutions.

Owing to the solubility of the precipitate in water, very dilute solutions must be evaporated nearly to dryness and cooled before the test is applied.

142. **Sodium hydrogen tartrate, $NaH\bar{T}$,** if it is added to some of the KCl solution in a test-tube, gives a white crystalline precipitate ($KH\bar{T}$). The precipitate usually appears only when the tube is well shaken for some time, after the mouth of the test-tube has been closed with the thumb.

Shake up the precipitate, and pour it off with the liquid into four test-tubes.

Add to three of these different portions, water, KHO, and HCl respectively; on being shaken or warmed with each of these liquids, the precipitate will dissolve.

To the fourth portion add alcohol; the precipitate will not dissolve.

Remarks.—When the above test is used for the detection of potassium, the precipitate will not be obtained at once in dilute solutions; but its formation is much hastened by the addition of alcohol. Very dilute solutions must first be concentrated by evaporation.

The solution to be tested should be neutral or feebly alkaline in reaction. If it is strongly acid in reaction, the free acid should be neutralised by the addition of sodium carbonate (Na_2CO_3) solution; or, if the acid is volatile, it may be removed by evaporation. Feebly-acid solutions may be at once mixed with NaHT. To an alkaline solution, tartaric acid (H_2T) should be added until the reaction of the liquid is strongly acid.

143. **Flame-Coloration (112).**—Dip a loop of platinum wire, which gives no colour to the flame, into some strong KCl solution; or moisten the wire, and dip it into some finely powdered KCl: then hold the loop in the flame. A *pale-lilac* or *lavender* coloration will be seen, if the KCl is pure. Even if the flame appears yellow to the naked eye, it will always become deep *crimson* when it is viewed through the thicker parts of an indigo-prism (Exp. 93, **112**).

The flame-coloration, due to potassium, shows in the spectroscope a *red* line (α) and a faint *indigo-blue* line (β) at the extreme ends of the spectrum (fig. 68, **116**). A faint continuous spectrum is also visible.

144. **Heat a little Solid KCl** in a small dry test-tube; or powder it finely, and heat it on a piece of platinum-foil. The KCl will melt, but it will only produce slight white fumes when it is heated very strongly. This proves that potassium salts are very slightly volatile even at a bright red heat.

The fused residue is often transparent and invisible; but its presence may always be detected by a slight crackling being heard, during the cooling of the foil or of the test-tube.

AMMONIUM (NH_4).—Use Ammonium chloride, NH_4Cl or AmCl.

145. **Potassium hydrate, KHO,** if it is poured in excess into some of the solution, or upon the solid, and heated, causes ammonia gas to be given off:

$$NH_4Cl + KHO = NH_3 + KCl + H_2O,$$

The ammonia is recognised:

(a) By its pungent smell, which is that of common *smelling salts.*

(b) By turning moistened red litmus-paper blue, or turmeric-paper brown.

Wet a small piece of the test-paper with distilled water, and place it upon the end of a glass rod. Then hold the rod in the test-tube, taking great care not to bring it into contact with the liquid or with the sides of the tube. The change of colour mentioned above will take place.

(c) By yielding white fumes with an acid gas or vapour. Hence if a glass rod or stopper, wetted with *strong* HCl, HNO_3, or $H\bar{A}$, is held over the mouth of the test-tube, dense white fumes will be seen.

146. **Platinum chloride, $PtCl_4$,** when it is stirred on a watch-glass with some AmCl solution, gives a yellow crystalline precipitate (Am_2PtCl_6).

This precipitate is soluble in water and in KHO solution, but is insoluble in alcohol. If it is boiled with KHO, NH_3 is evolved, and it is distinguished in this way from the similar potassium precipitate, K_2PtCl_6. The remarks in small type in paragraph 141 apply to this reaction also.

147. **Sodium hydrogen tartrate, $NaH\bar{T}$,** when it is shaken for some time in a test-tube with AmCl solution, gives a white crystalline precipitate ($AmH\bar{T}$).

This precipitate is soluble in water, in KHO, and in HCl, but is insoluble in alcohol. The remarks in small type in paragraph 142 apply to this reaction also.

148. **Flame-Coloration.**—Pure AmCl gives no flame-coloration. But since ammonium salts almost invariably contain a trace of sodium, they usually impart a yellow colour to the flame.

149. **Heat a little Solid AmCl** on a piece of platinum-foil. It will be converted into vapour and will produce

copious white fumes. These fumes are best seen when the foil is removed for a moment from the flame. The solid at last entirely disappears, showing that AmCl is completely volatile.

Repeat the experiment by heating a small piece of solid AmCl in a dry test-tube. The solid can be completely volatilised, and its vapour condensed as a white coating or *sublimate* on the upper part of the tube.

Some ammonium salts, such as the carbonate and the nitrate, are decomposed by heat into gases; they are therefore volatilised without producing white fumes or a sublimate.

SODIUM (Na).—Use Sodium chloride, NaCl.

150. Flame-Coloration.—NaCl gives an *intense yellow* colour to the flame. The colour is either invisible, or appears only pale blue, when it is viewed through the indigo-prism; *it never shows the slightest tinge of red* when it is seen through the thicker parts of the prism.

When the sodium coloration is examined by a small spectroscope, it gives a single yellow line *a* (fig. 68, **116**).

The yellow coloration of the flame which is produced by sodium is usually readily seen, even in the presence of other flame-colorations, since it is not easily masked. In any case the yellow spectrum line may always be detected, even when mere traces of sodium are present.

If both potassium and sodium are present, the potassium coloration is usually unseen, and the yellow coloration of sodium alone is visible. But when the flame is viewed through the indigo-prism, the crimson coloration of potassium is at once seen (**112**, Exp. 93). The lines of Na and K may also be distinctly separated from one another by means of the spectroscope.

151. Heat a little Solid NaCl in a dry test-tube or on platinum-foil. It will melt without producing white fumes, and will give slight fumes only when it is heated very strongly, showing that NaCl is very slightly volatile even at a bright red heat.

MAGNESIUM (Mg).—Use Magnesium sulphate, $MgSO_4.7H_2O$.

The $7H_2O$ in this formula represents the *water of crystallisation*, which is always present in the solid state in the crystallised salt. Water of crystallisation is entered in this way throughout the book.

152. Add Ammonium chloride (AmCl), then Ammonium hydrate (AmHO), and then **Sodium phosphate, Na_2HPO_4** : a white crystalline precipitate ($MgAmPO_4.6H_2O$) will form:—

$$MgSO_4 + AmHO + Na_2HPO_4 = MgAmPO_4 + Na_2SO_4 + H_2O.$$

If much water is present this precipitate will form only when the liquid is warmed, or is well stirred or shaken. It is soluble in HCl and in other acids, but is insoluble in AmHO.

153. **Potassium hydrate, KHO**, gives a white precipitate (MgH_2O_2), which is readily soluble in acids.

154. **Ammonium hydrate, AmHO**, gives a white precipitate (MgH_2O_2) : but if AmCl is added before the AmHO, no precipitate is produced.

155. **Ammonium carbonate, Am_2CO_3**, gives a white precipitate ($MgCO_3$), but only in strong solutions and on standing for some time. The previous addition of AmCl prevents the formation of this precipitate.

156. **Flame-Coloration.**—Pure $MgSO_4$ solution gives no colour to the flame.

157. **Blowpipe-test.**—If a little solid $MgSO_4.7H_2O$ is placed in a small cavity on a piece of wood-charcoal, and is then heated in the tip of the outer blowpipe-flame (110), it will shine brightly and will give a white unmelted mass of MgO. No white fumes will be given off, since MgO is not volatile.

If this white mass is allowed to cool and is moistened with several drops of cobalt nitrate solution, and is then again heated to whiteness for some time in the outer blowpipe-flame, it will assume a *delicate pink colour.* This colour will be readily seen when a piece of white paper is held near the cold residue on the charcoal.

DETECTION OF ONE METAL IN GROUP V., WHEN IT OCCURS ALONE.

The Detection of a Single Metal leads the beginner by easy stages to the detection of metals in the presence of one another. But the directions for the detection of a single metal, which follow the reactions in each Group, and the whole of Section V. which is concerned with the Analysis of Simple Salts, may be omitted if time must be saved.

After the student has carefully worked through the reactions for the metals in the Fifth Group, he should proceed to apply the experience, which he has thus acquired, to the detection of one metal belonging to this Group.

For this purpose a salt which contains only one of these metals should be supplied; and separate portions of the salt, or of its solution in water, should be subjected to the tests which are given in the first column of the following Table of Differences (160).

158. Explanation and Use of the Table of Differences.— A glance at this Table (160) will show that those tests have been selected, which serve to distinguish the metals of this Group from one another. The order in which the tests are placed is that which will best serve this object.

It will be evident that it is never necessary to try the whole of the tests upon any one substance. The presence of the metal, when it has been detected by one test, should however be confirmed by another test selected for that purpose. The third and fourth tests are also manifestly alternative ones, the use of $NaH\overline{T}$ being generally preferred to that of $PtCl_4$ for economical reasons.

A selection is made from the first four tests in examining a liquid. A solid substance should be first subjected to the fifth and sixth tests; and a portion of the solid should then be dissolved in water and the solution examined by the earlier tests.

As each test is made, the result which it yields is carefully observed. Occasionally no change is noticed; this is indicated by a blank in the column to the right. If any result is noticed, it is looked for in the columns to the right; it will indicate the presence of that metal, which heads the column in which the result stands.

Sodium is seldom entirely absent from any substance. The quantity present may be judged from the intensity of the yellow flame-coloration. Unless the coloration is very intense, the presence of a trace of sodium should be noted down, and another metal should be looked for.

The student will naturally take advantage of the aid afforded by the spectroscope in examining the flame-coloration (113–117).

Several simple unknown salts of the metals of Group V. should be examined by the Table (160), and the results obtained, together with the inferences drawn, should be carefully entered in the note-book.

159. Example of Entry in the Note-Book :—

A solid white substance was given to be tested for one metal in Group V.

Test.	Observation.	Inference
1 Heated in an ignition-tube.	Substance fused, but did not volatilise.	Absence of NH_4.
2. Heated on charcoal in the blowpipe-flame	Substance fused, and sank into the charcoal	Presence of K or Na
Dissolved part of the substance in water		
3. Held a drop on a platinum-wire loop in the flame.	A pale lilac flame coloration, which appeared crimson through the indigo-prism.	Presence of K.
4. Stirred a little of the solution with a few drops of $PtCl_4$ on a watch-glass.	A yellow precipitate formed.	Presence of K.
		Found K.

160. GROUP V.—TABLE OF DIFFERENCES.

Tests.	K—salts.	NH_4—salts.	Na—salts.	Mg—salts.
For solutions.				
1. Boil with KHO solution:	—	NH_3 gas given off. known by its smell and by turning moist turmeric brown, it also gives dense white fumes with strong HCl	—	White precipitate. Confirm by adding AmCl, AmHO, and Na_2HPO_4, to a fresh part of the solution (152)
2. Heat in the Bunsen-flame on a clean platinum wire:	*Pale lilac flame*, which appears crimson through the indigo-prism.	—	*Intense yellow* flame, which shows no tinge of red through the indigo-prism.	—
3. Add $PtCl_4$ make slightly acid with dilute HCl, and stir vigorously:	Yellow crystalline precipitate, forms only in strong solutions.	Yellow crystalline precipitate, forms only in strong solutions.	—	—
4. Add $NaH\overline{T}$ and shake well·	White crystalline precipitate in strong solutions.	White crystalline precipitate in strong solutions.	—	—
For solids only.				
5. Heat to redness in a dry test-tube, or on platinum foil:	Non-volatile, unless heated to bright redness	Volatile; the substance passes, at least partially, into vapour it usually gives white fumes, and forms a sublimate in the upper part of the tube.	Non-volatile, unless heated to bright redness	Non-volatile.
6 Heat strongly on charcoal in the outer blowpipe-flame:	Easily fusible, sinking into the charcoal, and colouring the flame pale lilac. (See 2.)	Volatile, giving off white fumes	Easily fusible, sinking into the charcoal, and colouring the flame intense yellow. (See 2.)	White infusible residue is left, which if moistened with $Co(NO_3)_2$ solution and reheated becomes pink.

DETECTION OF METALS IN GROUP V., WHEN THEY OCCUR TOGETHER.

Paragraphs, the numbers of which are marked with an asterisk (*), may be omitted by students who are learning the analysis of Simple Salts only.

161.* **When more than One Metal** of the Fifth Group is present in the substance to be examined, the possibility of interference in the reactions arises. It should be remembered, however, that—

1. NH_4 may always be detected by the evolution of NH_3 when the substance is boiled with KHO solution.

2. Na can always be found by its yellow flame-coloration.

3. Mg is indicated with certainty by its precipitation by KHO solution, and by Na_2HPO_4 in the presence of AmCl and AmHO.

4. K may always be found by the flame-coloration appearing crimson when it is viewed through the indigo-prism, or by the red line in its spectrum.

5. The test for K by means of $PtCl_4$ or $NaH\bar{T}$ is only conclusive in the absence of NH_4. If NH_4 is present, its salts must be removed by ignition, before K can be tested for by precipitation.

162.* The student should proceed to examine by the following Table (**163**) several solutions of unknown composition, containing two or more metals of this Group. Only one of the observations which are recorded against each test will be made. In future Tables the bracketed paragraphs, which concern the absence of the metals, will be omitted; but the student should invariably enter his negative results, together with the inferences which he draws from them.

The experiments, observations, and inferences should be entered in the note-book fully in the form shown in the Table (**163**).

163.* TABLE FOR TESTING FOR Mg, K, Na, NH$_4$, IN A SOLUTION WHICH MAY CONTAIN ALL OF THEM.

Experiment.	Observation.	Inference.
I. Add to a small part of the solution AmCl, then AmHO and Na$_2$HPO$_4$, shake well and allow to stand if no precipitate appears at once.	1. A white precipitate forms.	Presence of Mg.
	[2. No precipitate appears.]	[Absence of Mg.]
II. Dip a small loop of clean platinum wire into the solution, and hold it near the top of the Bunsen-flame.	1. A yellow flame.	Presence of Na, possibly also of K.
	Look at the yellow flame through the indigo-prism, a crimson red colour is seen.	Presence of K.
	2. A pale lilac flame, appearing crimson red when seen through the indigo-prism.	Presence of K and absence of Na.
	[3. No colour is imparted to the flame.]	[Absence of K and Na.]
III. Boil some of the solution in a test-tube with KHO solution, and try whether NH$_3$ is given off by smelling; or if it cannot be smelt, by testing with moistened turmeric-paper or strong HCl.	1. NH$_3$ gas is smelt, or the turmeric becomes brown, and white fumes are obtained with HCl.	Presence of NH$_4$.
	[2. No NH$_3$ is smelt, and the turmeric-paper does not become brown, neither are white fumes obtained with HCl.]	[Absence of NH$_4$.]

IV. A confirmatory test for K may be made by adding PtCl$_4$ or $NaH\overline{T}$. If Exp. III. has shown that NH$_4$ is present, proceed to test for K by column A; if NH$_4$ is absent, test for K by column B (161, 5).

A	B
Boil some more of the solution down to dryness in a porcelain dish, scrape out the white substance left in the dish, and heat it strongly on a piece of platinum-foil as long as any white fumes are seen to be given off when the foil is taken out of the flame for an instant. Place the foil when cold in a test-tube, and boil it with *very little* water, to which a drop of dilute HCl has been added, and evaporate to several drops; cool the liquid, and pour it out upon a watch-glass, add a little PtCl$_4$, and stir well:— A yellow precipitate.—*Presence of K.*	Pour a little of the solution upon a watch-glass, add PtCl$_4$, and stir well: a yellow precipitate:—*Presence of K.* If no precipitate forms, K may nevertheless be present, the solution being too dilute to allow the precipitate to form; therefore pour some of the solution into a porcelain dish, boil down nearly to dryness, cool and test this liquid by stirring it with PtCl$_4$ on a watch-glass, adding alcohol if the precipitate does not appear at once: a yellow precipitate:—*Presence of K.* *Note.*—Addition of alcohol will frequently produce the precipitate without evaporation.

REACTIONS OF THE RARER METALS IN GROUP V.

The rarer metals included in this Group are Li, Rb, Cs.

LITHIUM (Li).—Use Lithium chloride, LiCl.

164. Li occurs frequently in mineral waters and in the ashes of plants, also in small quantities in the minerals lepidolite, triphane, and petallite

It is allied to the metals of the Barium Group by the difficult solubility of its carbonate and phosphate ; and it differs from K and NH_4 by not being precipitated by $PtCl_4$ or by $H_2\overline{T}$; from Na it is readily distinguished by its flame-coloration and spectrum.

LiCl may be separated from KCl and NaCl by its insolubility in a mixture of absolute alcohol and ether.

165. **Sodium Phosphate, Na_2HPO_4,** if it is added to the solution, which must not be too dilute and must be made strongly alkaline with NaHO, will give, on boiling, a white crystalline precipitate (Li_3PO_4) which settles quickly. Traces of Li may be precipitated by adding Na_2HPO_4, and then NaHO until the liquid remains alkaline, evaporating to dryness and washing the residue with dilute AmHO.

This precipitate may be distinguished from the phosphates of Ba, Sr, Ca, and Mg by heating it on charcoal in the blowpipe-flame, when it will readily fuse and be absorbed by the charcoal support. The diluted cold solution of Li_3PO_4 in HCl also gives no precipitate when AmHO is added in excess, but a white crystalline precipitate forms when the liquid is boiled.

166. **Flame-Coloration**: this is *carmine-red*; the spectrum (fig. 68, 116) consists of an intense carmine-red line (α).

The flame-coloration is concealed by that of Na, but the presence of Na does not interfere with the spectrum. The Na-coloration is removed if the flame is viewed through the thinner parts of the prism.

The lithium flame-coloration differs from that given by K by being either unable to penetrate the thick layers of indigo-solution, or by being much lessened in intensity by passage through the indigo.

Lithium silicate only gives the flame-coloration after it has been fused with $CaSO_4$; lithium phosphate requires to be first moistened with HCl.

RUBIDIUM (Rb), CÆSIUM (Cs).—Use RbCl and CsCl.

167. The metals Rb, Cs occur in small quantities in some mineral waters and in a few minerals.

The compounds of Rb and Cs resemble those of K by being precipitated by $PtCl_4$ and by $H_2\overline{T}$, and by giving a flame-coloration similar to that of K

These metals differ from K by the greater insolubility in water of their platino-chlorides. K_2PtCl_6 can be dissolved away from Rb_2PtCl_6 and Cs_2PtCl_6 by boiling the precipitates with successive small quantities of water The alums also show a similar difference in solubility in cold water.

168. **Flame-Colorations.**—The flame-colorations given by these metals are not distinguishable from that given by K. Cs and Rb are, however, readily distinguished from one another and from other elements by their spectra (fig. 68, 116). The blue lines (α, β), given by Cs, are especially distinct and characteristic. In the Rb-spectrum the indigo-blue lines (α, β) are very distinct, but the red lines (γ, δ) are most characteristic. The chlorides are the most suitable salts for the spectroscope test.

DETECTION OF LI, RB, CS IN ANALYSIS.

The metals Li, Rb, Cs are most readily detected in this Group by means of their spectra.

For the detection of these metals in the ordinary course of analysis refer to the fifth column of the Table in paragraph 1090.

GROUP IV.—BARIUM GROUP.

169. This group includes Ba, Sr, Ca.

These metals differ from those of Group V. by being precipitated from their solutions as carbonates by ammonium carbonate, and as oxalates by ammonium oxalate, in the presence of ammonium chloride. They are not precipitated by any other Group-reagent.

BARIUM (Ba).—Use Barium chloride, $BaCl_2.2H_2O$.

170. **Ammonium carbonate, Am_2CO_3,** added after AmCl, causes a white precipitate ($BaCO_3$). This precipitate is at first flocculent; but it slowly shrinks in volume and becomes crystalline, if the liquid which contains it is gently heated and is then allowed to stand. Prove that this precipitate is soluble in acetic acid ($H\bar{A}$).

171. **Calcium sulphate, $CaSO_4$, or Sulphuric acid, H_2SO_4,** causes a heavy white precipitate ($BaSO_4$), which is insoluble in HCl even when the liquid is boiled.

172. **Potassium chromate, K_2CrO_4,** added after some $H\bar{A}$, produces a yellow precipitate ($BaCrO_4$) which is soluble in warm HCl, but differs from $SrCrO_4$ by being insoluble in warm $H\bar{A}$.

173. **Hydrofluosilicic acid, H_2SiF_6,** produces a semi-transparent precipitate ($BaSiF_6$). The precipitate does not appear in dilute solutions until after some time; its formation is hastened by boiling, shaking, or stirring the liquid, or by adding alcohol. H_2SiF_6 yields no precipitates with solutions containing Sr or Ca.

174. **Ammonium oxalate, $Am_2C_2O_4$,** produces a white precipitate (BaC_2O_4).

175. **Flame-Coloration:** $BaCl_2$ gives a yellowish-green

colour to the flame, which is visible through the indigo-prism.

The barium-spectrum (fig. 68, **116**) consists of a number of lines, the most characteristic of which are three green lines, α, β, γ.

STRONTIUM (Sr).—Use Strontium nitrate $Sr(NO_3)_2.4H_2O$.

176. Am_2CO_3, added after some AmCl, gives a white precipitate ($SrCO_3$), which is soluble in $H\bar{A}$. This precipitate is flocculent at first; but it gradually becomes crystalline and shrinks very considerably at ordinary temperatures, and more rapidly when it is heated.

177. $CaSO_4$, or **H_2SO_4**, gives a white precipitate ($SrSO_4$). This precipitate does not usually form in a cold solution at once, but only after some time. The precipitate, however, *appears at once when the liquid is boiled.*

178. **K_2CrO_4**, added after some $H\bar{A}$, causes no precipitate, since $SrCrO_4$ is soluble in $H\bar{A}$.

179. **$Am_2C_2O_4$** produces a white precipitate (SrC_2O_4).

180. Flame-Coloration: crimson-red; this colour appears *intense red* through the indigo-prism, unless the flame-coloration is very faint.

The strontium-spectrum contains many lines (fig. 68, **116**). The most characteristic are the orange line (α), the red lines (β, γ), and the blue line (δ).

CALCIUM (Ca).—Use Calcium chloride, $CaCl_2.6H_2O$.

181. Am_2CO_3, added after AmCl, gives a white precipitate ($CaCO_3$) which is soluble in $H\bar{A}$. This precipitate is flocculent at first; but it shrinks considerably and becomes crystalline after a time, more rapidly when it is gently heated.

182. $CaSO_4$: no precipitate, even when the liquid is allowed to stand, or is boiled.

183. H_2SO_4: a white precipitate ($CaSO_4$) forms at once in strong solutions, and often in weak solutions when they are boiled; but some $CaSO_4$ will always remain dissolved, since it is not quite insoluble in water.

Prove this by boiling the liquid which contains the precipitate, and then filtering it. Then keep adding AmHO to the filtrate and stirring it, until a drop of the solution turns turmeric-paper brown and the liquid smells of NH_3. Then add $Am_2C_2O_4$; a white precipitate will form, showing the presence of Ca (185) which must have been dissolved as $CaSO_4$

184. **K_2CrO_4**: no precipitate.

185. $Am_2C_2O_4$: white precipitate (CaC_2O_4), soluble in most acids, but insoluble in $H\bar{A}$ and in oxalic acid ($H_2C_2O_4$).

186. Flame-Coloration: yellowish-red. When this coloration is viewed through the indigo-prism it *does not appear red*, and differs in this respect from that given by Sr.

In the calcium-spectrum (fig. 68, 116) the most characteristic lines are the green line (β) and the intense orange line (α).

DETECTION OF ONE METAL IN GROUP IV., WHEN IT OCCURS ALONE.

187. The following Table of Differences will serve to detect a single metal in Group IV., when all other metals are absent. For an explanation of the method of using the Table, the remarks on the use of the corresponding Table for Group V. may be consulted (158).

GROUP IV.—TABLE OF DIFFERENCES.

The first three tests only need be used for the detection of a member of this Group when it occurs by itself in solution : the first test serves also for solids.

The flame-coloration obtained in Exp. 1 should be examined by the spectroscope.

Tests	Ba—salts	Sr—salts.	Ca—salts
1 Flame-coloration :	Greenish-yellow.	Intense crimson, appearing *crimson* through the indigo-prism.	Yellowish-red, not appearing red through the indigo-prism
2 $CaSO_4$ added to the cold solution :	An immediate precipitate.	A precipitate appearing only after a short time, *or immediately on boiling*.	No precipitate even on boiling
3 K_2CrO_4 added after $H\bar{A}$:	Light yellow precipitate	——	——
[4 H_2SiF_6 added	White precipitate	——	——]
5. H_2SO_4 added in excess and boiled :	*Entirely* precipitated as $BaSO_4$.	*Entirely* precipitated as $SrSO_4$	*Partially* precipitated as $CaSO_4$ (183)

SEPARATION AND DETECTION OF THE METALS IN GROUP IV.,

WHEN THEY OCCUR TOGETHER.

188.* The following Differences presented by the solutions of these metals are made use of :—

1. Their different flame-colorations, which are examined if necessary through the indigo-prism and by means of the spectroscope.

2. The difference in the behaviour of their solutions with $CaSO_4$ solution, which at once indicates the presence or absence of Ba, or of Sr in the absence of Ba.

3. The insolubility of $BaCrO_4$ in $H\bar{A}$, and hence the possibility of separating Ba, if present, by adding $H\bar{A}$ and then K_2CrO_4.

4. The fact that Sr, but not Ca, can be precipitated by $CaSO_4$ on boiling. This reagent will therefore indicate the presence of Sr in the absence, or after the separation, of Ba.

5. The complete precipitation of Sr, if present, by boiling the solution with H_2SO_4; sufficient $CaSO_4$ still remaining in the filtrate, to give a precipitate on the addition of $Am_2C_2O_4$ and of AmHO in excess.

189.* A Solution which has to be Examined for Ba, Sr, and Ca, and which can contain only these substances, is first made alkaline, if it is not already so, by the addition of AmHO in excess (**103**).

Am_2CO_3 is then added, and the addition of this reagent is continued as long as it causes any further precipitate after the liquid has been warmed, stirred well and allowed to settle. The liquid is then filtered. A little more Am_2CO_3 is added to the clear filtrate, and if this causes any further precipitate more Am_2CO_3 is added, and the liquid is again poured through the same filter.

As soon as the filtrate gives no further precipitate with Am_2CO_3, all the metals of this Group, which were present in the solution, will have been precipitated as carbonates, and will remain on the filter after the filtration of the liquid.

The precipitate is then examined by Table IV. (**1032**, **1033**), or by the simpler Table IV.A. (**1034**).

GROUP III.—IRON AND ZINC GROUPS.

The Group includes Al, Fe, Cr, Zn, Mn, Ni, Co, together with the rarer metals U, In, Be, Ti, V, Zr, Ce, Ta, Nb, La, Di, Y, E, Th. It is conveniently subdivided into Groups III.A. and III.B.

The reactions of the rarer metals are given in paragraphs 243-276.

GROUP III.A.—IRON GROUP.

190. This Group includes Al, Fe, Cr.

The members of this Group differ from those of Groups III.B., IV., and V. by being precipitated from their solutions by AmHO after the addition of AmCl. They are not precipitated, however, by the group-reagents for Groups II. and I. This Group is also precipitated by Am_2S, or by H_2S added to the alkaline solution.

Group III.A. further differs from Group III.B. in being completely precipitated by adding $BaCO_3$ shaken up with water. This reagent affords the most perfect means of separating these two Sub-groups from one another.

The members of Group III.A. show no characteristic flame-colorations; but, with the exception of Al, they impart a characteristic colour to the borax-bead.

ALUMINIUM (Al).—Use Aluminium sulphate, $Al_2(SO_4)_3$.

191. AmHO: white flocculent precipitate ($Al_2H_6O_6$), which is seen best after the liquid has been boiled. It is soluble in HCl and in $H\bar{A}$, and is slightly soluble in AmHO in the absence of AmCl:—

$$Al_2(SO_4)_3 + 6AmHO = Al_2H_6O_6 + 3Am_2SO_4.$$

192. KHO added in very small quantity: white flocculent precipitate ($Al_2H_6O_6$), soluble in excess of KHO. Add dilute HCl gradually to part of the KHO solution of the precipitate until the liquid is neutral, the $Al_2H_6O_6$ is reprecipitated. If more acid is added the precipitate dissolves, but it reappears when AmHO is added in slight excess.

From another portion of the KHO solution of the precipitate the $Al_2H_6O_6$ may be reprecipitated by the addition of sufficient AmCl solution.

193. **Ammonium sulphide, Am_2S**: the same precipitate ($Al_2H_6O_6$), H_2S gas being evolved, or remaining dissolved in the liquid:—

$$Al_2(SO_4)_3 + 3Am_2S + 6H_2O = Al_2H_6O_6 + 3Am_2SO_4 + 3H_2S.$$

194. **Blowpipe-test.**—Heat some solid $Al_2(SO_4)_3$ on charcoal in the outer blowpipe-flame; then moisten it, after cooling, with $Co(NO_3)_2$ solution and again heat it in the outer flame; a *fine blue mass* will be obtained.

IRON (Fe).—Use Ferric chloride, Fe_2Cl_6, and Ferrous sulphate, $FeSO_4.7H_2O$.

Ferric salts are usually yellow or reddish-yellow in colour. **Ferrous salts** are usually pale green if they are hydrated, but white if they are perfectly anhydrous.

Use Fe_2Cl_6 solution and $FeSO_4$ solution for reactions **196–198**, and solid $FeSO_4.7H_2O$ for **199** and **200**. In preparing $FeSO_4$ solution, crush a crystal of the solid substance and shake it in a test-tube with *cold* water.

195. **Two classes of Iron compounds** are known, which differ in appearance and in properties and behave differently with reagents. They are distinguished as *ferrous* and *ferric* compounds respectively.

In the former class the metal may be termed *ferrosum* (Fe″), and in the latter *ferricum* (Fe‴). The dashes following the symbol denote the number of Cl atoms, or their equivalent, which are combined with one atom of Fe in its compounds.

It is usual in stating analytical results to mention in which state of combination the iron exists, and therefore some distinctive tests are described below.

The whole of the reactions of ferrous compounds are not given, because Fe is always separated and detected in the course of analysis as a ferric compound, and ferrous compounds are subsequently tested for by trying special tests upon the original substance.

Ferrous compounds are readily converted into ferric compounds by boiling them for a short time with a little strong HNO_3, or with HCl and a crystal of $KClO_3$.

Both classes of compounds give the same results in the blowpipe-reactions (199, 200).

196. **AmHO**, or **KHO** : with ferric salt a reddish-brown flocculent precipitate ($Fe_2H_6O_6$), insoluble in KHO, soluble in HCl: with ferrous salt a dingy green precipitate, ultimately turning to brown $Fe_2H_6O_6$ on exposure to the air.

197. **Am_2S** : a black precipitate (FeS), which is mixed with white sulphur in the case of ferric salts :—

$$Fe_2Cl_6 + 3Am_2S = \overline{2FeS + S} + 6AmCl.$$

FeS is soluble in boiling acids, but is insoluble in KHO.

In very dilute iron-solution a green colour only is produced at first by Am_2S, but black FeS separates after the liquid has stood for some time or when it is boiled.

198. **Hydrogen sulphide, H_2S**, either passed as gas into the solution, or added as H_2S water, gives a black precipitate (FeS) when the iron-solution is subsequently mixed with excess of alkaline hydrate.

In neutral or acid solution of ferric salt, sulphur is precipitated and the ferric salt is converted into ferrous salt; the colour of the solution therefore changes from yellow to pale green : this change of colour is visible in a strong solution, after it has been boiled and filtered :—

$$Fe_2Cl_6 + H_2S = 2FeCl_2 + 2HCl + S.$$

199. **Heated with Solid Sodium carbonate (Na_2CO_3) on Charcoal** in the inner blowpipe-flame, a solid iron compound leaves a grey powder of metallic iron (110). This is sepa-

rated by levigation (111), and is shown to be *magnetic* by being attracted when it is touched under water with the end of a magnet, or with the point of a magnetised knife-blade.

200. **A Borax-bead** (108) containing Fe is *reddish-brown* while hot and *yellow* when cold, after it has been fused in the outer blowpipe-flame: after fusion in the inner-flame it is *olive-green*.

FURTHER DISTINCTIVE TESTS FOR FERROUS AND FERRIC SALTS.

201. For these tests the Fe solution should first be acidified by the addition of several drops of dilute HCl.

Reagents to be added.	Ferric-salts (Use Fe_2Cl_6 solution)	Ferrous-salts (Use $FeSO_4$ solution)
1. Potassium ferrocyanide (K_4FeCy_6):	*Dark blue precipitate*, "Prussian blue", insoluble in HCl, and turned brown by KHO	*Light blue precipitate*, becoming dark blue in the air, or on addition of HNO_3 or of Br-water.
2. Potassium ferricyanide (K_3FeCy_6). *	*No precipitate* the solution darkens, but is seen to contain no precipitate when it has been diluted with water.	*Dark blue precipitate*, "Turnbull's blue"; insoluble in HCl In *very* dilute solution only a dark bluish-grey colour is produced.
3. Potassium sulphocyanide (KCyS):	*Blood-red coloration*. no precipitate is produced, the liquid being perfectly clear on dilution, the colour is immediately destroyed when several drops are poured into some $HgCl_2$ solution its production is hindered by HNO_3 and by $H\bar{A}$	*No red coloration* unless small quantities of ferric-salts are present.

* **NOTE.**—This solution must be made immediately before it is used, by dissolving a small piece of solid potassium ferricyanide, about the size of a pin's head, in a few drops of water.

It will be seen that the addition of KOH or of AmOH (196) also serves to distinguish ferrous from ferric salt, when these salts are not present together.

CHROMIUM (Cr).—Use Chrome-alum, $CrK(SO_4)_2.12H_2O$, which contains $Cr_2(SO_4)_3$.

Chromic salts are usually bluish-green or violet in colour.

Remarks.—Chromium forms two classes of compounds. In one class chromium in combination with oxygen acts as an acid-radicle. This class includes the chromates, which are usually yellow or reddish in colour.

But chromium also forms a series of salts in which it acts as a metallic radicle; these are usually green or violet, and give the following reactions for Cr

The chromium salts pass by oxidation into chromates in reactions 204, 206, where the oxidation is caused by PbO_2 and by KNO_3 respectively.

Chromates, on the other hand, pass by reduction into green chromic compounds; examples of this change will be found under the tests for chromates (592).

202. **AmHO:** pale bluish-green or purple precipitate ($Cr_2H_6O_6$). If AmHO is added in large quantity and the liquid is heated, some of the precipitate will dissolve, producing a beautiful violet-red solution. The colour of the liquid is best seen after the precipitate has been removed from it by filtration. From this red solution the $Cr_2H_6O_6$ is reprecipitated when the liquid is boiled for several minutes in a porcelain dish.

203. **KHO**, if it is added in small quantity, gives the same precipitate ($Cr_2H_6O_6$). If more *cold* KHO is added, the precipitate is entirely dissolved to a green fluid. When this liquid is diluted with water and then boiled for several minutes, the $Cr_2H_6O_6$ is reprecipitated and the liquid becomes colourless. When AmCl is added to the KHO solution, the $Cr_2H_6O_6$ is also reprecipitated.

204. **Lead peroxide** (PbO_2), **or Red lead,** may be added to the green liquid which has been obtained by adding KHO in excess to the chromium solution (203). When this liquid is boiled with the lead oxide, its colour changes to yellow; a yellow precipitate ($PbCrO_4$) may then be thrown down from this yellow solution by adding to it $H\bar{A}$ in excess.

205. **Am_2S** also precipitates $Cr_2H_6O_6$, H_2S gas being given off or remaining dissolved. See the equation in paragraph 193.

206. A Solid substance containing Cr, if it is heated with fused (Na_2CO_3) and potassium nitrate (KNO_3) on platinum or porcelain, yields yellow sodium chromate (Na_2CrO_4). See Exp. 85 (106). When the cold product is dissolved in boiling water, a yellow liquid is obtained. If this solution is acidified with $H\bar{A}$, and is then boiled for a few minutes in order to drive off CO_2, it gives with solution of lead acetate ($Pb\bar{A}_2$) a yellow precipitate of $PbCrO_4$.

207. A Borax-bead containing Cr (108) is green after it has been fused either in the outer or in the inner blowpipe-flame.

DETECTION OF ONE METAL IN GROUP III.A., WHEN IT OCCURS ALONE.

208. The following Table of Differences will serve to detect one metal in Group III.A., when all other metals are absent. Refer to the remarks on the corresponding Table for Group V. (158), for an explanation of the method of using this Table.

GROUP III.A.—TABLE OF DIFFERENCES.

Tests.	Al—salts	Fe'''—salts	Fe''— salts.	**Cr—salts**
For liquids				
1. Add AmHO.	*White* flocculent precipitate	*Reddish brown* flocculent precipitate	*Dingy green* precipitate, rapidly turning brown when left exposed to the air	***Pale green*** **flocculent precipitate colour unaltered by exposure to air**
2. Add KHO.	*White* flocculent precipitate, *easily soluble in excess of KHO not reprecipitated on diluting and boiling for some time*	*Reddish brown* flocculent precipitate, *insoluble in excess of KHO*	The same precipitate as with AmHO, *insoluble in excess of KHO*	***Pale green*** **precipitate,** ***soluble in excess of cold KHO, but reprecipitated on diluting and boiling for some time.***
3. Add KCyS and a few drops of HCl:	——	*Deep blood red* coloration, destroyed by pouring into $HgCl_2$ solution	***No coloration*** **unless Fe''' is also present.**	——
Blowpipe tests for solids				
4. Fuse with Na_2CO_3 and KNO_3 on platinum foil	——	On cooling, a white mass of Na_2CO_3 remains, with dark brown particles of Fe_2O_3	Same as Fe'''	**On cooling, a** ***yellow*** **mass remains; the solution in $H\bar{A}$, gives a yellow precipitate with $Pb\bar{A}_2$**
5. Heated on charcoal:	If moistened with $Co(NO_3)_2$ solution and reheated strongly gives a *fine blue mass.*	In inner flame mixed with Na_2CO_3 gives a grey magnetic powder.	Same as Fe'''.	——
6. Fused in borax bead:	——	*Reddish yellow* in outer flame *Greenish yellow* in inner flame	Same as Fe'''	***Green*** **both in outer and inner flames.**

SEPARATION AND DETECTION OF THE METALS IN GROUP III.A., WHEN THEY OCCUR TOGETHER.

209.* The following Differences are utilised :—

1. The solubility of $Al_2H_6O_6$ in *boiling* KHO, in which $Fe_2H_6O_6$ and $Cr_2H_6O_6$ are insoluble.

2. The conversion of $Cr_2H_6O_6$ by fusion with Na_2CO_3 and KNO_3 into soluble Na_2CrO_4· $Fe_2H_6O_6$, when it is thus treated, remaining as Fe_2O_3 which is insoluble in water.

3. Cr is then detected by the yellow colour of the aqueous solution of the resulting Na_2CrO_4, and by the formation of a yellow precipitate when this solution is acidified with $H\bar{A}$, and $Pb\bar{A}_2$ solution is added to it.

4. Fe is found by the blood-red coloration which is obtained, when KCyS is added to the solution in HCl of the Fe_2O_3 resulting from (2).

210.* A Solution which may contain Al, Fe, Cr, is examined in the following manner :—

(a) To a few drops of the liquid, acidified with HCl, a little K_4FeCy_6 is added; if a blue precipitate is produced, Fe is present; proceed to **(b)**: if no blue precipitate forms, proceed to (c). This portion of the liquid is rejected.

(b) Two other small portions of the acidified solution are then tested; one with freshly made solution of K_3FeCy_6, which, by giving a dark blue precipitate, would show the presence of Fe''; the other with KCyS, which may cause a blood-red colour, proving the presence of Fe'''. These portions of the liquid are rejected. Proceed to **(c)**.

(c) The rest of the original solution is boiled, after a few drops of strong HNO_3 have been added if ferrous salt is present. Some AmCl is added; then AmHO is gradually added, with constant stirring, until the liquid is just alkaline and smells faintly of NH_3. The liquid is then boiled and filtered, and the precipitate is examined by Table III.A. (**1027**), all the appended Notes being disregarded.

GROUP III.B.—ZINC GROUP.

211. This Group includes Zn, Mn, Ni, Co.

Compounds of these metals differ from those of metals of Groups IV. and V. by being precipitated from their neutral or alkaline solutions by ammonium sulphide (Am_2S), and from their alkaline solutions by H_2S They are not precipitated by the Group-reagents for Groups III.A., II., and I. Mn is, however, gradually precipitated by oxidation, if its solution is mixed with AmCl and excess of AmHO and is then exposed to the air.

The compounds of the metals of this Group show no characteristic flame-colorations; but, with the exception of those of Zn, they give characteristic colours to a borax-bead.

Finely powdered barium carbonate ($BaCO_3$) does not precipitate the metals of this Group from their solutions, as it does the metals of Group III.A., unless they are present as sulphates.

Note.—The *yellow* ammonium sulphide, which is repeatedly referred to in the tests for Ni and Co, is the reagent which is ordinarily found in the laboratory. It is a polysulphide, and is represented by the general formula Am_2S^n. The monosulphide, Am_2S or AmHS is colourless.

ZINC (Zn).—Use Zinc sulphate, $ZnSO_4.7H_2O$.

Zinc salts are usually colourless.

212. Ammonium sulphide, Am_2S: white precipitate (ZnS). This precipitate often appears yellow, owing to the presence of excess of yellow ammonium polysulphide (Am_2S_n) in the liquid. The true colour is evident, as soon as the precipitate, produced by Am_2S_n, has been separated from the liquid by settling or by filtration.

The colour and the solubility of this precipitate is proved in the next paragraph.

213. Hydrogen sulphide, H_2S: white precipitate (ZnS). Zn is only partly precipitated from the neutral solution by

H_2S, since H_2SO_4, in which ZnS is soluble, is formed during the reaction:

$$ZnSO_4 + H_2S = ZnS + H_2SO_4.$$

If, however, NaHO or AmHO has been added in sufficient excess to the zinc solution, it will form a clear solution (214, 215); and since free H_2SO_4 cannot exist in this alkaline liquid, the Zn may be entirely precipitated from it as sulphide by H_2S.

The addition of a sufficient quantity of solution of sodium acetate ($Na\bar{A}$) to the zinc solution also enables H_2S to effect complete precipitation of the Zn as sulphide, since acetic acid ($H\bar{A}$), in which ZnS is insoluble, is produced in the solution:

$$ZnSO_4 + H_2S + 2NaA = ZnS + Na_2SO_4 + 2H\bar{A}.$$

Add to some $ZnSO_4$ solution AmCl, then AmHO until it is alkaline, and then H_2S. White ZnS will be precipitated. Add to separate portions of this liquid HCl, $H\bar{A}$, and Am_2S respectively; ZnS will be found to be soluble in HCl, but insoluble in $H\bar{A}$ and in Am_2S.

Note.—The tests which show the solubility of the sulphides of the metals of this Group are best tried on the precipitate which is obtained by the addition of AmCl, AmHO in excess, and H_2S to the solution of the metal, unless freshly prepared *colourless* Am_2S can be obtained, since the *yellow* polysulphide solution causes sulphur to precipitate together with the metallic sulphide:

$$ZnSO_4 + Am_2S_2 = ZnS + S + Am_2SO_4.$$

The sulphur, thus precipitated, is not dissolved by the solvents of the metallic sulphide

214. KHO, added in small quantity, gives a white precipitate (ZnH_2O_2). The addition of more KHO causes this precipitate to dissolve, but it is formed again when much water is added and the solution is boiled. The Zn may also be precipitated from the KHO solution as ZnS by the addition of H_2S.

215. AmHO, if added in small quantity, also yields a precipitate, which is soluble in excess. The formation of this precipitate is prevented by the previous addition of AmCl.

216. Blowpipe-test on Charcoal.—Mix some finely powdered $ZnSO_4.7H_2O$ and Na_2CO_3 in a small cavity on wood-charcoal, and heat the mixture in the inner blowpipe-flame. An incrustation will be obtained on the charcoal, which is *yellow* while hot and *white* when cold. This deposit cannot be driven away by the outer blowpipe-flame, but it is easily removed when it is heated in the inner flame.

Moisten the cold incrustation with cobalt nitrate solution and heat it strongly in the outer blowpipe-flame; it will become *green*.

Note.—The above changes of colour are more distinctly seen when a little zinc oxide (ZnO) is heated in an ignition tube, or in the outer blowpipe-flame on charcoal. The oxide will be *yellow* while it is hot and *white* when it has cooled. If the oxide is moistened on charcoal with $Co(NO_3)_2$ solution, and is then reheated in the outer blowpipe-flame, it will become *green*.

MANGANESE (Mn).—Use Manganese chloride ($MnCl_2$), or Manganese sulphate ($MnSO_4$).

Manganous salts are usually of a *pale pink* colour. Alkaline manganates are *green*, permanganates are *purple*.

217. Am_2S: flesh-coloured or pale pink precipitate (MnS). The true colour of this precipitate is only seen after it has been separated by filtration, if the ammonium sulphide was yellow. The precipitate occasionally becomes dark green after standing for some time. The colour of the moist filtered precipitate always changes by oxidation to dark brown, when it is allowed to stand in contact with the air. For the solubility of this precipitate, refer to paragraph **218**.

218. H_2S precipitates pale pink MnS, partially from neutral solutions and entirely from alkaline solutions, but it produces no precipitate in the presence of HCl or of $H\bar{A}$.

Add to some $MnCl_2$ solution $AmCl$, $AmHO$ in excess, and then H_2S; and show with separate portions of the precipitate that MnS is soluble in HCl and in HĀ, but is insoluble in Am_2S.

219. KHO: white precipitate (MnH_2O_2), insoluble in excess, and quickly turning brown by oxidation in the air. This change of colour is most rapid after the precipitate has been filtered off, since it is then exposed more fully to oxidation by the air.

220. AmHO gives the same precipitate (MnH_2O_2). If sufficient AmCl is first added, however, AmHO produces no immediate precipitate; but if this alkaline liquid is exposed to the air, it gradually turns brown by oxidation, and the Mn is ultimately precipitated as a brown hydroxide.

221. Blowpipe-test by Oxidation.—When any solid substance containing Mn is heated *upon platinum foil* in the outer blowpipe-flame, in contact with a fused mixture of Na_2CO_3 and KNO_3, the product yields a *bluish-green mass* when it cools. The mixture of the substance with the alkaline salts should be heated by allowing the tip of the blowpipe-flame to touch the *under surface* of the foil, and the flame must not be allowed to come into contact with the surface of the mixture (**106**, Exp. 85).

The test may also be made by fusing a mixture of Na_2CO_3 and KNO_3 into a bead in a loop of platinum wire, then taking up a little of the manganese compound upon the surface of the moistened bead, and fusing it into the bead by heating it in the extreme tip of the outer blowpipe-flame.

This reaction is a very delicate test for the presence of manganese.

222. Blowpipe-test on Charcoal.—When any manganese compound is heated with fused Na_2CO_2 on charcoal in the inner blowpipe-flame it yields a grey magnetic powder of Mn (110, 111).

223. A Borax-bead (108), which contains *very little* Mn, will be *violet-red* whilst hot and *amethyst-red* when cool, after it has been fused in the outer blowpipe-flame. When the bead is heated in the inner blowpipe-flame it becomes *colourless*.

This reaction is very delicate, and yields a result with even minute quantities of manganese compounds. Larger quantities of manganese render the bead opaque, after it has been heated in the outer blowpipe-flame.

NICKEL (Ni).—Use Nickel sulphate, $NiSO_4.7H_2O$.

Nickel salts are usually *bright green* in colour.

224. Am_2S: black precipitate (NiS).

Add *yellow* ammonium polysulphide in excess, boil and filter. The filtrate will be coloured brown by NiS, which has been dissolved by the excess of the polysulphide.

Pour this dark-coloured filtrate into a porcelain dish and boil it for some time, adding distilled water if necessary in order to prevent the liquid from evaporating to dryness. The dissolved NiS will be reprecipitated, and when it has been filtered off, the filtrate will be colourless.

If $H\bar{A}$ is added in excess to the dark-coloured filtrate, the dissolved NiS will also be reprecipitated.

For the solubility of NiS, refer to the next paragraph.

225. H_2S: black precipitate (NiS) in a neutral solution, or in a solution which contains no free acid except $H\bar{A}$, (**213**). HCl and other acids prevent the precipitation.

Add to $NiSO_4$ solution AmCl, then AmHO in excess and then H_2S; and show with the precipitate thus produced, that NiS is insoluble in cold HCl and in $H\bar{A}$, but is dissolved when it is heated with HCl and a crystal of potassium chlorate ($KClO_3$).

226. KHO : light green precipitate (NiH_2O_2).

227. AmHO, added in very small quantity, gives a bluish-green precipitate (NiH_2O_2). This precipitate is soluble in excess of AmHO to a violet-blue liquid: it is also soluble in AmCl.

If AmCl is first added to the $NiSO_4$ solution, the addition of AmHO causes no precipitate.

228. Solution of Potassium cyanide (KCy), if it is freshly-prepared by dissolving a fragment of solid KCy in water, and is added in small quantity to the nickel solution, gives a yellowish-green precipitate ($NiCy_2$). The further addition of the KCy solution dissolves this precipitate, but the precipitate appears again when sufficient HCl is added to this solution.

229. Precipitation as $Ni_2H_6O_6$ from $NiCy_2.2KCy$.—Acidify some $NiSO_4$ solution with several drops of $H\bar{A}$; then add freshly-made KCy solution gradually, while stirring or shaking the nickel solution, until the precipitate, which forms at first, is just redissolved.

Boil this solution for a short time: then cool it, and divide it into two parts.

The addition of HCl in excess to one part will produce a precipitate of $NiCy_2$, which often appears only after a time.

A black precipitate ($Ni_2H_6O_6$) will be formed on the addition to the other part, either of sodium hypochlorite solution (NaClO), or of potassium hypobromite (KBrO). The hypobromite is conveniently prepared by adding to the nickel cyanide solution first KHO in excess, and then bromine water (Br) until the liquid remains yellow. The black precipitate forms more rapidly if the liquid is warmed.

230. Blowpipe-test on Charcoal.—When a mixture of finely-powdered $NiSO_4.7H_2O$ and Na_2CO_3 is heated on charcoal in the inner blowpipe-flame, a grey powder of Ni will remain, which may be separated by levigation and shown to be attracted by the magnet (110, 111).

231. A Borax-bead containing Ni is *violet* or *reddish-brown* immediately after it has been fused in the outer blowpipe-flame, and it becomes *pale yellow* on cooling.

If the bead is heated for some time in the inner blowpipe-flame, it becomes *black* and *opaque*.

COBALT (Co).—Use Cobalt nitrate, $Co(NO_3)_2.6H_2O$.

Hydrated cobalt salts are usually *reddish-pink* in colour; in the anhydrous condition they are usually *blue* or *purple*.

232. **Am_2S :** black precipitate (CoS). If much *yellow* ammonium polysulphide is added, and the liquid is then boiled and filtered, the filtrate is coloured *yellow* by Am_2S_n; it is *not dark coloured*, since CoS, unlike NiS, is insoluble in yellow Am_2S_n. For the solubility of CoS refer to the next paragraph.

233. **H_2S :** black precipitate (CoS), forming only in alkaline solutions, or in solutions which contain no free acid except $H\bar{A}$ (**213**). The presence of HCl prevents the precipitation.

Add AmCl, then AmHO in excess, and then H_2S to some $Co(NO_3)_2$ solution, and pour into two separate portions of this liquid HCl and $H\bar{A}$ respectively; the precipitate will not dissolve. To the portion containing HCl add a crystal of $KClO_3$ and heat; the precipitate will dissolve readily.

234. **KHO:** blue precipitate (CoH_2O_2). The colour of the precipitate changes by oxidation on exposure to the air, also when the liquid containing the precipitate is heated.

235. **AmHO :** bluish-green precipitate, having the same properties as that produced by KHO. The precipitate is soluble in excess of AmHO and in AmCl, and is therefore not formed if AmCl is added to the cobalt-solution before AmHO, or if AmHO is added in excess.

236. **Freshly-prepared KCy solution,** if it is added in small quantity, gives a reddish-brown precipitate ($CoCy_2$). When more KCy solution is added slowly and while shaking the liquid, the precipitate dissolves; but it reappears when HCl is added to the solution.

237. **Cobalticyanide reaction.**—Acidify some $Co(NO_3)_2$

solution with a few drops of $H\bar{A}$; then add freshly-made KCy solution slowly, until the precipitate which forms at first is just redissolved.

Boil this liquid, then cool it and divide it into two parts. Add to one of these portions HCl; and to the other either NaClO solution, or excess of NaHO solution followed by sufficient Br-water to render the liquid permanently yellow; no precipitate will appear in either case, even when the liquid is warmed. Refer to 229.

The difference in behaviour of solutions of Ni and of Co, which have been boiled with excess of KCy, is due to the fact that $NiCy_2$ forms with KCy a compound ($NiCy_2 2KCy$) which is easily decomposed: whereas $CoCy_2$ forms with excess of KCy, in the presence of atmospheric oxygen, the very stable compound K_3CoCy_6, *potassium cobalticyanide,* which resists decomposition by acids and by alkaline oxidising solutions —

$$2CoCy_2 + 8KCy + 2H_2O + O = 2K_3CoCy_6 + 2KOH + H_2O.$$

238. **Blowpipe-test on Charcoal.**—When a solid cobalt compound is mixed with Na_2CO_3, and the mixture is fused on charcoal in the inner blowpipe-flame, it yields a grey magnetic powder of Co(110,111).

239. **A Borax-bead,** which has been dipped into a strong solution of $Co(NO_3)_2$, assumes a *fine blue* colour when it is fused in either the inner or the outer blowpipe-flame. This colour is produced even by minute quantities of cobalt compounds.

DETECTION OF ONE METAL IN GROUP III.B., WHEN IT OCCURS ALONE.

240. **The Table of Differences** on pp. 150-1 will serve to detect one metal in Group III.B. when all other metals are absent.

The distinction between Ni and Co in the Table depends upon noting the presence of NiS in *yellow* ammonium polysulphide; this is proved by a dark filtrate being obtained when the precipitated sulphide is warmed with excess of the yellow polysulphide and the liquid is filtered.

SEPARATION AND DETECTION OF THE METALS IN GROUP III.B., WHEN THEY OCCUR TOGETHER.

241.* The following Differences are made use of:—

1. The solubility of NiS in yellow ammonium polysulphide, in which ZnS, MnS and CoS are insoluble.

NiS may then be reprecipitated by boiling this solution, or by the addition of $H\bar{A}$, and the presence of Ni may be confirmed by heating the NiS in a fused borax-bead (231).

2. The solubility of ZnS and MnS in cold dilute HCl, in which NiS and CoS are almost insoluble.

3. The solubility of ZnH_2O_2 in cold KHO, in which MnH_2O_2 is insoluble.

White ZnS may then be precipitated from this alkaline solution by the addition of H_2S: and the MnH_2O_2 may be heated in a fused mixture of Na_2CO_3 and KNO_3, when it will yield a bluish-green mass (221).

4. The difference in behaviour of the solution of $NiCy_2$ and of $CoCy_2$ in excess of KCy solution, when the liquid is warmed with NaClO, or with NaBrO (229, 237).

5. The marked difference in colour of the borax-bead containing Ni from that containing Co is a further means of detecting these metals.

Mn may be separated from Zn, Ni, and Co by passing H_2S into the neutral solution which has been saturated with $Na\bar{A}$ (213); Mn alone remains in solution (218), and the other metals are precipitated as sulphides.

Another method for separating Ni and Co depends upon the fact that while Co is precipitated as $Co_2(HO)_6$ from its solution by $BaCO_3$ in the presence of Br, Ni is not precipitated by this means and remains in solution.

242.* A Solution is Examined for Zn, Mn, Co, Ni, by adding AmCl, then AmHO in excess, and then yellow

This Table runs
across both pages

GROUP III.B.

Tests.	Zn—salts.	Mn—salts.
For liquids.		
1. Add Am_2S; or better AmCl, AmHO in excess and H_2S-water.	*White precipitate*: soluble in cold dilute HCl: *insoluble in* $H\bar{A}$.	*Pink precipitate*: soluble in cold dilute HCl: *soluble in* $H\bar{A}$.
2. Add KHO:	*White precipitate: soluble in excess of KHO.*	*White precipitate*: turning brown in the air: *insoluble in KHO.*
3. Add KCy solution: This test need only be tried when a *black* precipitate has been obtained by Test No. 1	—	—
Blowpipe tests for solids.		
4. Fused with Na_2CO_3 on charcoal in the inner blowpipe flame.	A white incrustation, which, if moistened with $Co(NO_3)_2$ and heated in the outer flame, turns *green* The substance itself, when strongly heated after having been moistened with $Co(NO_3)_2$ also becomes green.	A grey powder. The substance, if fused with Na_2CO_3 and KNO_3 on platinum foil in outer flame, gives a *bluish green* mass.
5. Borax bead:	—	Outer flame —*Amethyst red.* Inner flame —*Colourless.*

Am_2S_n until the liquid smells of the sulphide after it has been well stirred or shaken.

The liquid is then heated, and a few drops of it are poured upon a filter. If the filtrate is colourless, sufficient Am_2S_n has not been added; more Am_2S_n must then be poured in, the liquid must be once more filtered, and the colour of the filtrate must be again observed.

As soon as the filtrate is brown or yellow in colour, proving that Am_2S_n has been added in excess, the whole of it is heated and filtered, and the precipitate is examined by Table III.B. (1028).

If the filtrate is yellow it is rejected, if it is brown it must be examined for Ni according to paragraph 1020.

TABLE OF DIFFERENCES.

Ni—salts.	Co—salts.
Black precipitate: soluble in boiling yellow Am_2S to a dark solution: insoluble in cold dilute HCl and in $H\bar{A}$. *Green precipitate*, insoluble in KHO. Added in slight excess to the feebly acid solution and boiled for some time, then Br-water and excess of NaHO added, gives a black precipitate on heating	*Black precipitate: insoluble in boiling yellow* Am_2S, in cold dilute HCl, and in $H\bar{A}$. *Blue precipitate:* insoluble in KHO. Added in slight excess to the feebly acid solution and boiled for some time, then Br-water and excess of NaHO added, gives no precipitate on heating.
A grey magnetic powder.	A grey magnetic powder.
Outer flame. *Violet* or *yellow* while hot. *Yellow* when cold. Inner flame.—*Grey* or *opaque* bead.	Outer and inner flames.—*Fine blue* bead.

REACTIONS OF THE RARER ELEMENTS IN GROUP III.

243. In this Group are included U, In, Ti, Be, Tl, [V], (Zr, Ce Ta, Nb, La, Di, Y, E, Th).

244. Tl is often partially precipitated as chloride in Group I., and its reactions are given under that Group. V is not precipitated unless acid is added in excess, after Am_2S has been added.

URANIUM (U).—Use Uranyl nitrate, $(UO_2)''(NO_3)_2$.

U occurs in nature principally as *pitchblende*, an oxide; also as *uranite*, a hydrated uranium calcium phosphate; and as *chalcolite*, hydrated uranium copper phosphate.

245. **AmHO, KHO, or NaHO**: yellow precipitate of uranate, insoluble in excess.

246. **Am_2S** gives in neutral solutions a *dingy yellow* or *brown* precipitate of uranium oxysulphide, which differs from ZnS, MnS, and FeS by being soluble in Am_2CO_3. The precipitated oxysulphide settles slowly, unless AmCl is added, it is soluble in acids, even in $H\bar{A}$; when it is heated with Am_2S in excess, it is changed into uranous oxide and sulphur.

247. **H_2S** produces no precipitate in acid solutions.

248. **Am_2CO_3, $KHCO_3$, or $NaHCO_3$**: yellow precipitate of double carbonate, easily soluble in excess; from this solution the uranium is reprecipitated by the addition of NaHO or KHO, or by boiling the liquid; it differs in this respect from the corresponding iron precipitate.

249. **K_4FeCy_6** gives in acid solutions a reddish-brown precipitate, which differs from the similar one produced in a copper solution, by dissolving in excess of AmHO to a *yellow* liquid.

250. **$BaCO_3$** causes complete precipitation even in the cold.

251. **Zn** changes the *yellow* colour of acid solutions to *green*.

252. **Borax and Microcosmic beads**: after being heated in the outer flame, *yellow*; inner flame, *green*.

INDIUM (In).—Use Indium sulphate, $In_2(SO_4)_3$.

Indium occurs in zinc-blende and in wolfram

253. **Alkaline hydrates** precipitate a hydrate resembling $Al_2H_6O_6$, which is insoluble in excess. Indium solutions are also precipitated by alkaline carbonates, by Na_2HPO_4, by boiling their neutral solutions with excess of $Na\bar{A}$, by $BaCO_3$, and by alkaline oxalates.

254. **H_2S** gives no precipitate in strong and acidified solutions; in dilute and feebly acid solutions a little sulphide separates; in a solution containing no free acid but $H\bar{A}$ the metal is entirely precipitated as *yellow* sulphide. This precipitate is insoluble in cold Am_2S, but dissolves when the Am_2S is boiled: *white* indium sulphide separates as the hot solution cools.

255. **Am_2S**, added after $H_2\bar{T}$ and excess of AmHO, gives a white precipitate, which becomes yellow when it is treated with $H\bar{A}$.

256. **Flame-Coloration**: bluish-violet. The spectrum shows a very

characteristic blue line; when indium chloride is introduced into the flame this line is brilliant, but it very rapidly disappears (fig. 68, 116).

BERYLLIUM (Be).—Use Beryllium sulphate, $Be_2(SO_4)_3$.

Be occurs as silicate in phenakite, and as silicate with aluminium silicate in beryl and in emerald.

257. **Am_2S, AmHO, KHO, or NaHO** precipitates the flocculent hydrate, which resembles $Al_2H_6O_6$ in appearance and in being soluble in KHO: it differs from $Al_2H_6O_6$ in being precipitated from its solution in KHO by dilution and long boiling, and by the freshly precipitated hydrate being dissolved when it is boiled for some time with AmCl solution.

258. **Alkaline carbonates** precipitate a carbonate, which is soluble in excess of the reagent; the carbonate is reprecipitated when this solution is diluted and boiled for some time; the solution and reprecipitation are most easily effected when Am_2CO_3 is used as the reagent. (Difference from $Al_2H_6O_6$.)

259. **$BaCO_3$** precipitates Be solutions completely.

260. **$H_2C_2O_4$ and Alkaline oxalates** produce no precipitate.

261. **Moistened with $Co(NO_3)_2$ solution and heated** on charcoal in the outer blowpipe-flame, a *grey* mass is obtained. (Difference from Al.)

TITANIUM (Ti).—Use Titanium oxide, TiO_2.

Ti occurs as TiO_2, with traces of Fe, Mn, Cr; also in rutile, anatase, and brookite. TiO_2 occurs also in combination with Fe in titaniferous iron ore.

262. **Ignited TiO_2** is insoluble in water and in most acids: it is easily soluble in HF, less readily in boiling strong H_2SO_4: it also becomes soluble in cold water after it has been heated with fused $KHSO_4$. TiO_2 differs from SiO_2 by not being volatilised when it is heated in a platinum dish with HF and strong H_2SO_4.

263. **By Dilution and long Boiling,** white flocculent hydrated TiO_2 is precipitated from solution in H_2SO_4 or in HCl, and from the aqueous solution prepared after treatment with fused $KHSO_4$: the precipitate is metatitanic acid: it cannot be filtered off until either an acid or AmCl has been added.

264. **AmHO, KHO, NaHO, Am_2S, or $BaCO_3$,** white flocculent precipitate, insoluble in excess ; if the precipitation and washing have been carried out in the cold, the precipitate dissolves in HCl and in dilute H_2SO_4.

265. **Zn or Sn** gives in strong acid solutions a *blue* coloration ; if the solution is dilute, a *rose-red* coloration is produced.

266. **$Na_2S_2O_3$** precipitates Ti solution entirely when the liquid is boiled. (Difference from Fe.)

267. **Microcosmic-bead**: in the outer flame *yellow* while hot, *colourless* when cold: in the inner flame *yellow* while hot, *violet* when cold. The production of these colours is promoted by introducing a fragment of Sn into the fused bead. The addition of a small quantity of $FeSO_4$ causes the bead to become *blood-red*, when it is heated in the inner blowpipe-flame.

VANADIUM (V).—Use Sodium vanadate, $NaVO_3$.

V occurs in vanadinite [$3Pb_3(PO_4)_2.PbCl_2$], and in certain Fe-ores and Cu-ores. Vanadium is known in several stages of oxidation ; it usually occurs in analysis as vanadic acid or a vanadate, which in acid solution is of a yellow or reddish colour.

271. **H_2S, H_2SO_3, or $H_2C_2O_4$** reduces an acid solution of a vanadate, and causes the colour of the solution to change to *blue ;* with H_2S a deposit of sulphur is also formed.

272. **Am_2S,** if added in excess, gives a brown liquid ; the addition of an excess of acid to this brown liquid produces a *brown* precipitate of V_2S_5 ; the brown precipitate is redissolved by an excess of Am_2S, yielding a reddish-brown liquid

273. **Zn,** when introduced into a very dilute solution, which is acidified with H_2SO_4 and is then gently warmed, causes the liquid to change in colour to *blue*, then to *green*, and then to *lavender-blue.*

274. **Solid AmCl,** if it is added to the solution until it is saturated, precipitates Am_3VO_4, which is insoluble in saturated AmCl solution.

275 **H_2O_2,** when shaken up with an acid solution, imparts to it a red colour ; the colour is not removed by shaking the liquid with ether. This is a very delicate test.

276. **Borax-bead**: in the outer flame *colourless*, or *yellow* if much V is present: in the inner flame *green* hot and cold, or, if much V is present, *brown* hot and *green* cold.

DETECTION OF THE RARER ELEMENTS IN GROUP III.

For the Detection of these Rarer Elements of Group III. in the ordinary course of analysis, refer to the third and fourth columns in Table 1090, and to Table 1091.

GROUP II.—COPPER AND ARSENIC GROUP.

This Group includes the metals Hg, Pb, Bi, Cu, Cd, As, Sb, Sn, together with the rarer elements Pd, Os, Rh, Ru, Au, Pt, Mo, Se, Te, Ir.

Group II. is subdivided into Groups II.A. and II.B.

The Reactions of the Rarer Elements are given in paragraphs 348–378.

GROUP II.A.—COPPER GROUP.

277. This Group includes Hg″, Pb, Bi, Cu, Cd, (Pd).

The compounds of these metals differ from those of the metals in Groups III.A., III.B., IV., and V. by being precipitated as sulphides by H_2S from their acid solutions. With the exception of Pb, which is partially precipitated from strong solutions as chloride, these metals are not precipitated from their solutions by HCl or by soluble chlorides.

The precipitated sulphides of the metals of Group II.A. differ from those of Group II.B. by being insoluble both in Am_2S and in KHO.

H_2S does not precipitate the members of this Group from strongly acid solution readily or completely, until the solution has been suitably diluted with water.

The Rarer Metal, Palladium (Pd), belongs to this Group; its reactions are given in paragraphs 348–351.

MERCURICUM (Hg″).—Use Mercuric chloride, $HgCl_2$.

278. Mercury forms two series of compounds, which are distinguished as *Mercuric* and *Mercurous* compounds. These two classes of compounds behave differently with reagents, and the metal present in them may be distinctively called *Mercuricum* and *Mercurosum*.

As Mercuricum the metal is classed in this Group, but as Mercurosum it is classed in Group I. In paragraphs 386 and 387 tests are given, which serve to distinguish mercury as Mercurosum from mercury as Mercuricum.

279. **Hydrogen sulphide, H_2S**: black precipitate (HgS). If the H_2S solution is added slowly, the precipitate is first *white*, then *brown* or *orange*, and ultimately *black*. The occurrence of these changes of colour, during the addition of H_2S, is characteristic of mercuric salts.

Filter off the precipitate of HgS, and wash it free from dissolved chlorides; then place portions of it in two test-tubes.

Boil one of these portions with strong HNO_3, the precipitate will not be dissolved; now add HCl and warm again, the precipitate will dissolve.

Boil the second portion with Am_2S, the precipitate will not dissolve.

Hence HgS is insoluble in hot strong HNO_3, and in Am_2S. It is, however, easily soluble in a mixture of HNO_3 and HCl, which is known as *aqua regia*.

280. **Stannous chloride, $SnCl_2$**; white precipitate (Hg_2Cl_2): if an excess of $SnCl_2$ is added, the white precipitate becomes grey: the grey precipitate consists of fine particles of Hg:—

$$2HgCl_2 + SnCl_2 = Hg_2Cl_2 + SnCl_4$$
$$Hg_2Cl_2 + SnCl_2 = 2Hg + SnCl_4.$$

Boil the liquid, and decant it from the grey precipitate. Then heat the precipitate with strong HCl; the Hg-particles will usually unite to globules, which are visible through a lens and are frequently seen even by the naked eye.

281. **Copper, Cu**: cleanse a small strip of copper sheet, or a copper coin, by rubbing it with sand-paper, or by dipping it into strong HNO_3. Immerse it in some $HgCl_2$ solution, which has been acidified by the addition of a few drops

of HCl. The Cu will become coated with a grey film of Hg :—

$$HgCl_2 + Cu = Hg + CuCl_2$$

Allow the Cu to remain immersed in the liquid for several minutes, then remove it, dry its surface, and rub it with a cloth or with a piece of wash-leather. The Cu will appear more or less whitened like silver, since the mercury has formed an *amalgam* with it.

The Hg may be readily driven off from the copper by heating it strongly. If the copper strip is heated in a test-tube, or better in a small hard-glass tube sealed at one end, a sublimate of globules of Hg will form upon the cool sides of the tube.

282. **Heating with Sodium carbonate, Na_2CO_3.**—Dry some sodium carbonate perfectly by heating it strongly on platinum foil or in a porcelain dish. Also dry the interior of a small test-tube by heating it in the flame, and then sucking out the moist air by a glass tube. Mix a *little* finely-powdered $HgCl_2$ intimately with about four times its measure of the dry and finely-powdered $NaCO_3$. Then transfer the mixture into the tube and cover it with a layer of the dry Na_2CO_3.

Now cleanse the inside of the tube perfectly, and proceed to heat the upper layer of Na_2CO_3 strongly : gradually extend the heat to the mixture below, while the upper layer is maintained at a high temperature. Hg-vapour will be evolved, and will condense in globules on the inside of the tube.

Sometimes the globules of metal are so small that they appear as a grey film only, or they are few and scattered and are not easily visible. In either case the minute globules may usually be detected by means of a pocket-lens, or they may be united into larger and visible globules by rubbing the inside of the tube with a glass rod. Even every minute globules may be seen, when they have been flattened by pressing them with a glass rod against the inside of the tube.

Remarks.—The powder is most easily transferred to the tube, by alternately scooping it up with the mouth of the tube, and tapping the tube upon the table so as to shake the powder to the bottom.

Or the powder may be poured upon a narrow strip of notepaper, which has been folded over into a trough, and the paper may be then pushed into the horizontal tube; if the tube is now held erect and tapped, the powder will be easily transferred to the bottom of the tube without soiling the glass.

Both the inside of the tube and the powder must be perfectly dry, else particles will obstinately adhere to the inside of the tube and interfere with the detection of the metallic globules.

The heating must never be commenced until the inside of the tube is perfectly clean. It is cleansed, if necessary, by means of a twisted slip of filter-paper or with a wooden match.

If any drops of water condense on the inside of the tube during the first stage of the heating, they must be removed by a piece of filter-paper. If, however, proper care has been taken in drying the powder, no moisture will be evolved from it. The escape of steam or gases from the powder is very undesirable, as it is likely to sweep uncondensed mercury vapour out of the tube.

283. **Heat a little Solid $HgCl_2$** in a tube closed at one end; the substance will sublime in white fumes, showing that mercury compounds are volatile.

LEAD (Pb).—Use Lead acetate, $Pb\bar{A}_2.3H_2O$, dissolved in dilute $H\bar{A}$.

284. **H_2S:** black precipitate (PbS). This precipitate is often *red* if much hydrochloric acid is present in the solution; but the red colour changes to black when water is added and H_2S is passed, or when H_2S-water is added.

Filter, and show with separate portions of the precipitate that PbS is insoluble in KHO and in Am_2S, but is soluble in boiling dilute HNO_3; also that it is converted by boiling strong HNO_3 into white insoluble $PbSO_4$.

285. **HCl:** white precipitate ($PbCl_2$), which forms only in cold and strong solutions. Boil the precipitate with sufficient water: it will dissolve: but the $PbCl_2$ will separate

again in slender crystals, when the hot solution is slowly cooled.

286. H_2SO_4 : white precipitate ($PbSO_4$). This precipitate is much less soluble in dilute H_2SO_4 and in alcohol than it is in water; hence the dilute H_2SO_4 should be added in excess to a pretty strong solution of the lead salt.

Shake up the liquid, and pour it off into two test-tubes; let the liquid stand in each of these tubes and decant it from the precipitate. Show that the precipitate may be dissolved by pouring upon it $H\bar{A}$, or $H_2\bar{T}$ solution, and then adding AmHO in excess and boiling; show also that it dissolves in boiling strong HCl.

287. K_2CrO_4, or $K_2Cr_2O_7$: yellow precipitate ($PbCrO_4$). Pour off the liquid with the precipitate into two tubes, and show that the precipitate is soluble in KHO, but insoluble in $H\bar{A}$.

288. Blowpipe-test on Charcoal.—Mix some powdered $Pb\bar{A}_2.3H_2O$ with about twice as much Na_2CO_3 in a cavity on a piece of charcoal, and heat the mixture in the inner blowpipe-flame (Exp. 88, **110**). A yellow incrustation will form upon the surface of the charcoal around the mixture, and small lustrous globules of Pb will be seen within the cavity. The incrustation is readily removed by heating it in the inner blowpipe-flame, and it colours the flame pale blue.

Detach one of the metallic globules with the point of a knife, place it on the flat bottom of the inverted mortar, and strike the globule a smart blow with the pestle. It will flatten out without cracking or breaking into powder, showing that Pb is *malleable* and not *brittle*.

If one of the cleansed globules is fixed upon the point of a penknife, and is then rubbed with gentle friction on paper, it will mark the paper as a blacklead pencil does.

Bismuth (Bi).—Use Bismuth nitrate, $Bi(NO_3)_3.5H_2O$, dissolved in dilute HNO_3.

289. H_2S : brownish-black precipitate (Bi_2S_3) :—

$$2Bi(NO_3)_3 + 3H_2S = Bi_2S_3 + 6HNO_3.$$

Decant most of the liquid from the precipitate; then shake up the precipitate with the remainder, pour some of the liquid with the precipitate into three test-tubes, and show that the precipitate is insoluble in Am_2S and in KHO, but soluble in strong HNO_3.

290. AmHO, or **KHO** : white precipitate (BiH_3O_3), insoluble in excess of the reagent. Pour off a small quantity of the liquid and precipitate into a test-tube, add much AmHO and warm : the precipitate will remain undissolved. Filter off the rest of the precipitate, and keep it for the reaction described in paragraph 291.

290a. **KHO and $SnCl_2$**. add KHO solution to $SnCl_2$ solution until the precipitate which forms at first is just redissolved, then add a few drops of $Bi(NO_3)_3$ solution : a black precipitate (BiO) will be formed.

291. H_2O.—Pour *a few drops* of boiling dilute HCl upon the precipitate which remained in the filter from par. 290, and allow the acid solution to drop from the funnel into a large quantity of distilled water. A *milkiness* will appear, which is due to the formation of BiOCl :—

$$BiCl_3 + H_2O = BiOCl + 2HCl.$$

Pour some of the milky liquid into two test-tubes ; add to one portion a little strong HCl and warm it, the precipitate will dissolve ; to the other portion add $H_2\bar{T}$ and warm, the milkiness will remain, differing in this way from that caused by SbOCl (328).

If Bi is present in small quantity only, this milkiness will often not appear until the liquid has been stirred and then allowed to stand for a few minutes.

A similar precipitate may be obtained by allowing the original bismuth nitrate solution to fall drop by drop into much water, but the nitrate is less completely precipitated than the chloride by mixture with excess of water.

292. **K_2CrO_4**, or **$K_2Cr_2O_7$**, added after some strong $Na\bar{A}$ solution, gives a yellow flocculent precipitate, $Bi_2(CrO_4)_3$. When KHO is added, the precipitate will not dissolve, differing in this way from $PbCrO_4$; it will dissolve, however, completely when HCl or HNO_3 is added in excess.

The addition of $Na\bar{A}$ in the above reaction is necessary in order to replace the nitric acid in the original solution by $H\bar{A}$, since the precipitate is readily soluble in HNO_3, but is insoluble in $H\bar{A}$:

$$Na\bar{A} + HNO_3 = NaNO_3 + H\bar{A}.$$

293. **H_2SO_4**: no precipitate: difference from Pb.

294. **Blowpipe-test on Charcoal.**—Heat a mixture of powdered $Bi(NO_3)_3$ and Na_2CO_3 in a cavity on charcoal in the inner blowpipe-flame. There will be seen around the cavity an incrustation which is *orange-red* while hot and *yellow* when cold. White globules of Bi will also be seen within the cavity; they are very brittle and will be easily crushed to powder by a sharp blow with the pestle; they are too hard to mark paper (288).

Copper (Cu).—Use copper sulphate, $CuSO_4.5H_2O$.

Two classes of copper salts are known, which are commonly distinguished as cupric and cuprous salts respectively.

Cupric salts are usually blue, but some are green; their colour commonly becomes very pale, or disappears, when the salts are thoroughly freed from water of crystallisation.

Cuprous salts are usually white or colourless. Their acid solutions become coloured in the air; they yield a precipitate of white Cu_2I_2

when they are mixed with KI solution, and yield red Cu_2O when they are mixed with KHO

Cupric oxide is *black*, cuprous oxide is *red*.

295. **H_2S**: brownish-black precipitate (CuS).

Filter, keeping the funnel carefully covered with a glass plate, since air quickly oxidises CuS to $CuSO_4$ and the $CuSO_4$ dissolves and runs through in the filtrate.

Place some of the precipitate in five test-tubes and show that it is insoluble in KHO, in Am_2S and in boiling dilute H_2SO_4, but that it is soluble in boiling HNO_3 and in KCy solution.

296. **AmHO**, if it is added in a diluted state and in very small quantity, gives a *greenish-blue* precipitate. If more AmHO is added this precipitate dissolves, yielding an *intensely blue* liquid. The solution becomes light blue again when an acid is added in excess.

297. **KHO** produces a pale blue flocculent precipitate, which becomes black when the liquid is heated.

297a. **H_2SO_4**: no precipitate: difference from Pb.

298. **Potassium ferrocyanide, K_4FeCy_6**: reddish-brown precipitate (Cu_2FeCy_6), insoluble in $H\bar{A}$; the colour of this precipitate is best seen when the reaction is tried in a white porcelain dish. In a *very dilute* copper solution only a reddish colour is seen at first when the ferrocyanide solution is added.

299. **Fe.**—Polish a piece of steel or iron wire or sheet, or the blade of a penknife, by rubbing it with sand-paper, remove all grease by boiling it in a little dilute KHO; then dip it into $CuSO_4$ solution, which has been acidified with a few drops of H_2SO_4; the immersed surface will be gradually covered with a red film of Cu.

300. **Zn and Pt.**—Place a piece of platinum foil or wire in

some $CuSO_4$ solution, which has been acidified with H_2SO_4 in a porcelain dish or in a watch-glass, then drop a small piece of Zn upon the platinum : a red film of Cu will be deposited upon the platinum.

301. **Blowpipe-test on Charcoal.** — If finely-powdered $CuSO_4.5H_2O$ and Na_2CO_3 are mixed in a cavity on charcoal, and the mixture is then heated in the inner blowpipe-flame, red scales or globules of Cu are obtained. These are best seen after they have been washed by levigation (111). The addition of solid KCy facilitates the smelting process and enables globules of copper to be obtained.

302. **A Borax-bead containing Cu** is *green* while hot and *blue* when cold, after it has been fused in the outer blowpipe-flame.

Fusion in the inner blowpipe-flame causes the bead to become *red;* it may even become *colourless*, if very little Cu is present and the bead is long heated in a good reducing flame: the introduction of a small fragment of Zn or Sn into the bead aids this change.

303. **Flame-Coloration.**—If a loop of platinum wire is dipped into $CuSO_4$ solution and is then held in the inner blowpipe-flame or in the Bunsen-flame, it imparts a *green* coloration to the flame.

The flame coloration shows a *blue centre* after the loop has been moistened with HCl, or if $CuCl_2$ solution was originally used.

CADMIUM (Cd).—Use Cadmium sulphate, $CdSO_4.4H_2O$.

304. **H_2S**: bright yellow precipitate (CdS).

Boil off the H_2S and pour the liquid containing the precipitate into five tubes.

Show with these separate portions that the precipitate is insoluble in Am_2S, in KHO and in KCy solution, but is soluble in boiling dilute HNO_3 and in boiling dilute H_2SO_4.

Dilute the H_2SO_4 solution of the precipitate with much water and pass H_2S for a short time: yellow CdS will be reprecipitated.

305. **AmHO**, if it is added in a very dilute condition and in small quantity, gives a white precipitate (CdH_2O_2); when more AmHO is added, the precipitate will be readily dissolved.

306. **H_2SO_4**: no precipitate: difference from Pb.

307. **Blowpipe-test on Charcoal.**—If powdered $CdSO_4$ is mixed with Na_2CO_3 in a cavity on charcoal, and the mixture is then heated in the inner blowpipe-flame, a brown incrustation will be seen on the surface of the charcoal around the cavity.

DETECTION OF ONE METAL IN GROUP II.A., WHEN IT OCCURS ALONE.

308. **The following Table of Differences** serves to detect a single metal in Group II.A., when all other metals are absent.

This Table runs
across both pages.

Group II.a.—

Tests.	Hg—salts.	Pb—salts.
For liquids		
1. Pass H_2S, or add H_2S-water:	*Black* precipitate; when filtered and well washed, insoluble in boiling strong HNO_3, and *unchanged* by it. Confirm by (281) in solution.	*Black* precipitate; almost insoluble in boiling strong HNO_3, changed by it into *white* $PbSO_4$
2. Add H_2SO_4:	——	White precipitate.
3 Add AmHO:	*White* precipitate, insoluble in excess, is usually obtained.	*White* precipitate, insoluble in excess
Blowpipe reactions for solids.		
4 Heated with Na_2CO_3 on charcoal in the inner blowpipe-flame	—— Test for Hg by (282)	*Yellow* incrustation; white *malleable* globules.

Separation and Detection of the Metals in Group II.a., when they occur together.

309.* **The following Differences** are utilised:—

1. The solubility of Bi_2S_3, CuS, and CdS in strong boiling HNO_3, in which HgS and PbS are insoluble. HgS and PbS remain as HgS and $PbSO_4$ respectively after the treatment with hot strong HNO_3.

2. The solubility of $PbSO_4$ in a mixture of $H_2\bar{T}$ with excess of AmHO, HgS being insoluble in this mixture.

3. The solubility of CuH_2O_2 and CdH_2O_2, and the insolubility of BiH_3O_3 in excess of AmHO.

4. The solubility of CdS, and the insolubility of CuS in boiling dilute H_2SO_4.

The insolubility of CdS in KCy solution, in which CuS easily dissolves, furnishes another means of separating Cu from Cd.

TABLE OF DIFFERENCES.

Bi—salts.	Cu—salts	Cd—salts.
Black precipitate; soluble in boiling strong HNO_3 to form a colourless solution.	*Black* precipitate; soluble in boiling concentrated HNO_3 to form a blue solution; insoluble in boiling dilute H_2SO_4.	*Yellow* precipitate; soluble in boiling HNO_3, and in boiling dilute H_2SO_4.
White precipitate, insoluble in excess. Confirm by (291).	*Blue* precipitate, easily soluble in excess to a deep blue liquid.	*White* precipitate, easily soluble in excess.
Orange red incrustation; white *brittle* globules.	No incrustation; red metallic scales Confirm by (302, 303).	*Brown* incrustation; no globules.

After each metal has been separated, its presence is then confirmed by some special test.

310.* A Solution which may contain the Metals of Group II.A. is first made acid, if it is not already so, by adding to it HCl. Whether the liquid is clear or not, H_2S is then passed through it for about five minutes, and the precipitated sulphides are filtered off.

The clear filtrate is either mixed with H_2S-water, or it is diluted with a little water and H_2S is passed through it again for a short time, in order to ascertain whether the metals have been completely precipitated from the original solution.

If no further precipitate is produced, the filtrate may be rejected. But in case H_2S causes further precipitation, the gas is passed for some time longer and the liquid is then poured again through the filter. The filtrate is rejected only when H_2S no longer produces any precipitate in it.

The precipitate is then examined by Table II. (1024) commencing at Group IIA. Copper Group (1025), and using only the left-hand side of the Table.

GROUP II.B.—ARSENIC GROUP.

311. This Group includes As, Sb, Sn.

The metals of this Group differ from metals of Group III.A., III.B., IV., and V., by being precipitated as sulphides by H_2S from a solution which has been acidified with HCl. They differ from metals of Group I. by not being precipitated by HCl.

The sulphides of the metals of this Group differ from those of the metals in Group II.A. by being soluble in Am_2S_n and in KHO.

Each metal of this Group forms two series of compounds. These compounds resemble each other in many reactions, but they present certain differences which serve to distinguish them. The two classes are conveniently distinguished by the terminations *-ic* and *-ous*, as in the two classes of mercury and copper salts: thus we speak of arsen*ic* acid and arseni*ous* acid, and of stann*ous* and stann*ic* chloride

The Reactions of the Rarer Elements of this Group, Au, Pt, Mo, Se, Te, are given in paragraphs 352–378.

ARSENICUM (As).—Use Arsenious oxide, As_2O_3, and Sodium arsenate, $Na_2HAsO_4.12H_2O$.

The Arseni*ous* compound, As_2O_3, is dissolved by boiling it for some time with water; the Arsen*ic* compound, $Na_2HAsO_4.12H_2O$, is also dissolved in water.

312. The tests described in paragraphs 313–321 give the same ultimate results with an arsen*ic* compound as with an arseni*ous* compound. As a rule, however, the reactions proceed slowly with an arsen*ic* compound, since the first stage consists in the conversion of the arsen*ic* compound into an arseni*ous* compound.

An arsen*ic* compound is converted into an arseni*ous* compound by boiling it with H_2SO_3 solution, with strong HCl, or with some other reducing agent. An arsen*ious* compound is converted into an arsen*ic* compound by heating it with HNO_3, or with some other oxidising agent.

313. **H_2S** produces only a yellow coloration in neutral solution of an *arsenious compound.* But if the solution is acidified with a few drops of HCl, a light yellow flocculent precipitate (As_2S_3) will form immediately even in the cold.

Filter off some of the As_2S_3, and show that it is insoluble in boiling strong HCl.

Pour some of the liquid containing the precipitate into two test-tubes, and add gradually KHO and Am_2S respectively; each of these solutions will completely dissolve the As_2S_3. The As_2S_3 may be reprecipitated from these solutions by the addition of HCl in excess.

In cold acidified solutions of *arsenic compounds* H_2S produces no precipitate. But if strong HCl is added and the solution is boiled, H_2S will produce a precipitate of white S and then one of yellow As_2S_3, when it is passed for some time into the boiling liquid.

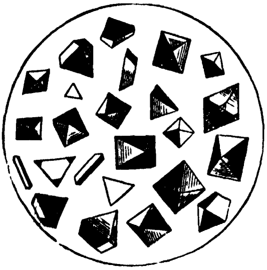
The complete precipitation of arsen*ic* solutions by H_2S is most rapidly effected, if they have been first converted into the arsen*ious* state. This may be effected by adding H_2SO_3 to the boiling arsen*ic* solution until it smells persistently of SO_2, and then boiling the liquid until SO_2 is no longer smelt. The arsenious compound thus produced may then be readily and completely precipitated by H_2S in the cold solution.

314. **Reinsch's Test.**—Several narrow strips of sheet copper are cleansed by means of sand-paper, or by dipping them into strong HNO_3. They are then boiled in the arsenious solution, which has been previously acidified by dilute HCl. The surface of the copper will soon be coated with a dark grey film of As_2Cu_5.

The strips are carefully removed from the liquid, rinsed, and gently pressed between filter-paper; they are then dried by heating them in the steam-oven, or by holding them in the fingers and heating them *gently* over the flame. The dry strips are then gradually heated over a very small flame in a warm and dry test-tube. A white sublimate of As_2O_3 will form on the upper part of the tube.

A much more characteristic sublimate will be obtained, if moisture is entirely removed from the copper strips and from the tube, and if the sublimation is then effected very slowly, and *while the sides of the tube are hot.* This sublimate will be sparkling and crystalline; and if the crystals are examined under the microscope, they will be seen to be transparent, colourless, regular octahedra and tetrahedra (fig. 69). The formation of these crystals under the above conditions is a most valuable confirmation of the presence of As.

FIG. 69.



ARSENIOUS OXIDE CRYSTALS.

The sublimate of As_2O_3 may be dissolved in a little boiling water, and the presence of As may be confirmed by applying tests 313 and 323 to this solution.

The presence of As in the original grey deposit upon the Cu should always be confirmed by one of the above methods, since other metals may be deposited as grey films on Cu.

Reinsch's test will serve for the detection of arsenic compounds, after their solutions have been reduced to arsenious compounds by being mixed with strong HCl or with H_2SO_3 solution and boiled.

TESTS WHICH DEPEND UPON THE FORMATION OF ARSENIC HYDRIDE (AsH_3).

The following tests (315–319), which depend upon the formation of *very poisonous* AsH_3 gas, should be performed in a closet provided with a good draught: this precaution is necessary in order to guard against any of the gas being inhaled.

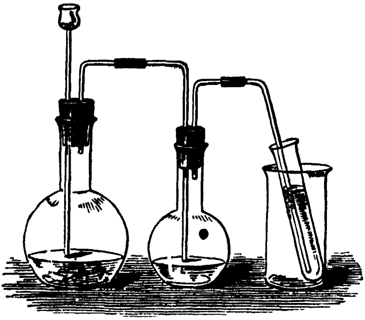
315. Hofmann's Test.—Pour dilute HCl upon some

Zn in a small hydrogen-flask, fitted as is shown in fig. 70.

The hydrogen may be washed, as it escapes, by letting it bubble through some water contained in the second flask, but this is not necessary.

Allow the hydrogen to pass through some $AgNO_3$ solution contained in a test-tube. If the Zn and acid are free from As, as they should be, no precipitate or colour will be produced in the $AgNO_3$ solution.

FIG. 70.



HOFMANN'S ARSENIC APPARATUS.

Now pour *a little* As_2O_3 solution down the funnel into the hydrogen flask; a black precipitate of Ag will form in the $AgNO_3$ solution.

This precipitate is produced by the AsH_3, which is formed by the action of the nascent hydrogen upon the As_2O_3:—

$$As_2O_3 + 12H = 2AsH_3 + 3H_2O.$$

The AsH_3, on passing into the $AgNO_3$ solution, reacts thus;—

$$AsH_3 + 6AgNO_3 + 3H_2O = 6Ag + H_3AsO_3 + 6HNO_3.$$

It will be seen that Ag is precipitated, while H_3AsO_3 remains in solution.

As soon as a copious precipitate has been obtained, the test-tube is removed.

It will be best to set aside the test-tube with its contents to be examined afterwards, and to proceed at once to use the gas, while it is being briskly evolved, for reactions 316 and 317.

The liquid in the test-tube is filtered; the precipitate is rejected, and several drops of $AgNO_3$ solution are added to the clear filtrate. Some very dilute solution of ammonia is made by pouring several drops of AmHO solution into a test-tube, then nearly filling up with distilled water and inverting the tube. When this solution is mixed drop by drop with the filtrate, a light yellow precipitate (Ag_3AsO_3 or Ag_2HAsO_3) will be obtained.

In order to explain the formation of this precipitate, it must be remembered that the solution contains H_3AsO_3 in the presence of $AgNO_3$ and of HNO_3: and further that $AgNO_3$ forms a yellow precipitate with H_3AsO_3 if no free acid is present in the liquid (323). The formation of this precipitate is prevented by the HNO_3; but when sufficient AmHO is added to neutralise the free HNO_3 which is already present, as well as that which is formed by the action of $AgNO_3$ on H_3AsO_3, the yellow precipitate will appear:

$$H_3AsO_3 + 3AgNO_3 + 3AmHO = Ag_3AsO_3 + 3AmNO_3 + 3H_2O$$

This precipitate (Ag_3AsO_3) is, however, readily soluble in AmHO; hence it is necessary to use *very dilute* AmHO and to add it gradually in order to avoid excess, since if excess of AmHO were added the precipitate would be prevented from forming.

316. **Marsh's Test.**—Disconnect the flasks (fig. 70), and slip into the rubber joint a tube of hard-glass about four inches in length, supporting it on the ring of a retort-stand (fig. 71). Then pour in a little strong HCl through the funnel-tube.

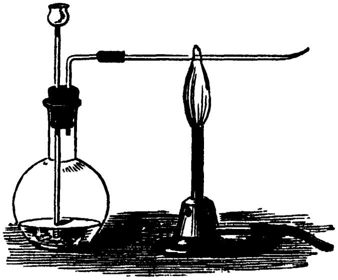
Caution.—If the experiment is performed as is here directed, the hydrogen will have been already evolved for some time for the previous test (315), and will have expelled all the air from the flask. There will therefore be no risk of an explosion occurring when the gas is lighted.

But if the hydrogen is being produced for the first time, or if the flask has been opened or recharged, allow the gas to escape briskly for four or five minutes; and then ascertain that a test-tube, filled with the gas, burns quietly, before proceeding to kindle the gas at the jet. In any case it is well to cover the flask with a cloth before the hydrogen is kindled, in order to guard against any risks from an explosion (34).

Now light the hydrogen as it escapes from the jet (fig. 71). The flame will be seen to differ from that of pure H by its livid blue colour, and by its evolution of white fumes of As_2O_3. If these appearances are not noticed, there is not sufficient arsenic solution in the flask. A little more of the solution must, therefore, be poured in through the funnel-tube.

Press down upon the flame *the inside* of a porcelain crucible lid or of a porcelain crucible or dish. A dusky black film of As will be deposited upon the cool surface. Two porcelain surfaces should be thus stained with As.

FIG. 71.



MARSH'S ARSENIC MIRROR.

Pour upon one of these stains some freshly made solution of bleaching-powder; the stain will be rapidly dissolved.

Warm the second stain of As with Am_2S. It will dissolve, and the solution will leave *yellow* As_2S_3 when it is slowly evaporated.

317. Decomposition of AsH_3 by Heat.—Pour a little more strong HCl into the flask (fig. 71), if necessary, in order to cause the hydrogen to come off rapidly. Light the gas at the jet, and heat a point near the middle of the tube strongly (fig. 71). A black mirror of As will form just beyond the part which is heated by the flame. The flame burning at the jet will meanwhile become colourless, showing that the arsenic has been removed from the hydrogen.

Heat another point in the tube, so as to produce a second mirror; then divide the tube between the mirrors.

Dissolve one mirror, when it is cold, by warming it with some fresh solution of bleaching-powder in a test-tube.

Heat the other mirror gently in a test-tube held obliquely in the flame; if the sides of the tube have been first heated, a crystalline sublimate of As_2O_3 will be obtained (fig. 69, p. 170). Dissolve the cold sublimate in a little hot water and confirm the presence of As by trying reactions 313 and 323 on the solution.

318. **$HgCl_2$ solution** is coloured by contact with AsH_3. Remove the cork from the larger flask (fig. 70, p. 171) and push a plug of cotton-wool into its neck, in order to stop any spirting frem the liquid in the flask. Then cover the mouth of the flask with a small piece of filter-paper, which has been moistened with $HgCl_2$ solution; the paper will become coloured, first *yellow* and then *brown*.

If paper moistened with $AgNO_3$ solution is substituted for the above paper, it will be *blackened* (315).

319. **Fleitmann's Test.**—Boil some pieces of Zn in KHO solution: H will be slowly evolved, and will be free from odour. If a little As_2O_3 solution is now added and the liquid is again boiled, a garlic odour will be perceptible; the odour will be caused by the evolution of AsH_3:—

$$\begin{cases} Zn + 2KHO = H_2 + ZnK_2O_2. \\ As_2O_3 + 12H = 2AsH_3 + 3H_2O. \end{cases}$$

If a small piece of filter-paper, which has been moistened with $AgNO_3$ solution, is placed on the mouth of the tube while the liquid is being boiled, the paper will be stained black by the separation of Ag (315).

An arsen*ic* compound must be reduced, by being boiled with H_2SO_3, before the above test is applied.

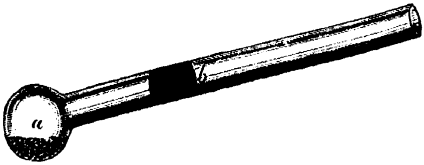
Sb compounds do not give the above result; the test therefore serves to distinguish between As and Sb.

REACTIONS OF SOLID ARSENIC COMPOUNDS.

320. **Mirror of As by Reduction.**—Dry some finely-powdered substance containing As thoroughly in the steam oven, and mix it intimately in a mortar with about four

times its measure of a freshly ignited mixture of KCy and Na_2CO_3. Heat this mixture in a little bulb-tube of hard glass (fig. 72), or in a narrow test-tube about three inches in length, carefully observing the precautions mentioned in paragraph **282.** A black mirror of As will form on the cooler part of the tube (*b*), and a smell of garlic may usually be detected at the mouth of the tube.

FIG. 72.



ARSENIC MIRROR FROM SOLID ARSENIC COMPOUND.

If the bulb is cut off and the mirror is gently heated in a test-tube, the As will be converted into a white sublimate of As_2O_3, or into transparent octahedral crystals of the oxide (fig. 69, p. 170).

321. Blowpipe-reaction on Charcoal.—Mix a solid substance, containing As, with Na_2CO_3 and KCy, and heat the mixture on charcoal in the inner blowpipe-flame. The flame will be coloured *livid blue*, unless this tint is overpowered by the yellow sodium coloration. A smell of garlic will be noticed when the charcoal is removed from the flame.

322. Heat a little Solid As_2O_3 in a small ignition-tube: it will be readily sublimed. If the As_2O_3 is perfectly dry and it is sublimed very slowly after the upper part of the tube has been first heated in the flame, the sublimate will sparkle, and when it is examined under the microscope, it will be seen to consist of octahedral crystals (fig. 69, p. 170).

DISTINCTIVE TESTS FOR ARSEN*IOUS* AND ARSEN*IC* COMPOUNDS.

Reagent.	Arsen*ious* compound Use As_2O_3 dissolved in H_2O.	Arsen*ic* compound Use $Na_2HAsO_4.12H_2O$ dissolved in H_2O
323. $AgNO_3$; several drops only are added.	*Light yellow precipitate* (Ag_2HAsO_3 or Ag_3AsO_3): this precipitate forms only when a little dilute AmHO is added carefully drop by drop * show that it is dissolved by AmHO and by HNO_3. The precipitate is also appreciably soluble in $AmNO_3$ hence, if too much AmHO has been added, the precipitate will not always form when the liquid is neutralised with HNO_3	*Brown precipitate* (Ag_3AsO_4): show that it is soluble in AmHO and in HNO_3.
324. $CuSO_4$; several drops only are added.	*Yellowish-green precipitate* ($Cu''HAsO_3$): this precipitate forms only when dilute AmHO is added carefully, drop by drop: * show that it is soluble in AmHO and in HNO_3.	*Pale green precipitate* ($CuHAsO_4$): soluble in AmHO and in HNO_3.
325. $CuSO_4$; two drops added, and then some KHO.	The above yellowish-green precipitate is first produced, but when more KHO is added the precipitate dissolves to a clear *blue* liquid, and this deposits *red* Cu_2O when it is boiled.	No clear blue solution is obtained, and no Cu_2O is produced when the liquid is boiled.
326. AmCl, AmHO, and $MgSO_4$:	No precipitate.	White crystalline precipitate ($MgAmAsO_4$): if this is filtered off, and then moistened with $AgNO_3$ solution on the filter, it assumes a *brown* colour (**323**).

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* When As_2O_3 is dissolved in water, it yields H_3AsO_3; and this acid cannot give the Ag_3AsO_3 precipitate when $AgNO_3$ is added, because this decomposition would lead to the formation of HNO_3, in which Ag_3AsO_3 is soluble. Hence the addition of AmHO is necessary in order to prevent the formation of nitric acid. But since the precipitate is also soluble in AmHo, this reagent must be added carefully in quantity only just sufficient to neutralise the acid. This is best effected by using *very dilute* AmHO, which has been made by pouring a few drops of AmHO into a test-tube, then filling the tube two-thirds with distilled water, closing it with the thumb, and inverting it for a moment. This dilute AmHO is to be added drop by drop, shaking or stirring the test solution after each addition, until the precipitate is obtained.

ANTIMONY (Sb).—Use Antimony chloride, $SbCl_3$, dissolved in dilute HCl.

Antimon*ic* and antimon*ious* compounds give results, which are ultimately identical, in the following reactions (327–335), with the exception of reaction 328. After these reactions, which are common to Sb in both its sets of compounds, two are given which are distinctive of the two classes of compounds (336, 337).

327. H_2S : orange-red precipitate (Sb_2S_3). Pour off the liquid with the precipitate into three tubes, and show that the precipitate is dissolved when it is warmed with pure KHO or with Am_2S, but is reprecipitated from these solutions when HCl is added in excess. Show also that the precipitate is soluble in boiling strong HCl.

328. **H_2O.**—When $SbCl_3$ solution is poured into much water, it yields a white precipitate or turbidity (SbOCl). This precipitate is redissolved, when HCl is added to the liquid and it is heated; it is also readily soluble in $H_2\bar{T}$. The solubility of SbOCl in $H_2\bar{T}$ distinguishes it from the similarly formed BiOCl precipitate (291).

•329. **Zn and Pt.**—If the acid solution of $SbCl_3$ is poured upon some platinum-foil in a porcelain dish, and a small piece of Zn is dropped upon the foil, the platinum will be rapidly coated with a black deposit of Sb. If the foil is rinsed with water and is then heated in boiling HCl, the stain will remain undissolved; but it will be rapidly removed by heating the foil in boiling HNO_3.

The formation of a slight grey or brown stain on the Pt is no evidence of the presence of Sb. The presence of Sb in the stain may be proved by dissolving it in a little boiling dilute HNO_3, diluting the cold solution, and then passing H_2S through the solution and allowing it to stand at rest for some time, when red Sb_2S_3 will settle down.

TESTS WHICH DEPEND UPON THE FORMATION OF ANTIMONY HYDRIDE.

SbH_3 may be generated by pouring Sb-solution into a hydrogen-apparatus, as is directed in paragraphs 315–318 for an arsenic solution. Antimony hydride (SbH_3) is evolved. This gas differs from AsH_3 by being free from smell and from poisonous properties, and also in the respects mentioned in paragraphs 330–333.

330. **If SbH_3 is passed into $AgNO_3$ solution** (315), it produces a black precipitate ($SbAg_3$):—

$$SbH_3 + 3AgNO_3 = SbAg_3 + 3HNO_3.$$

Filter off this precipitate, and reject the filtrate, which will contain no Sb.

Wash the precipitate of $SbAg_3$ four or five times with boiling water upon the filter. Then pour upon it boiling dilute $H_2\bar{T}$ solution, which will dissolve the Sb. Receive the liquid in a test-tube, boil it, and pour it once more upon the filter. Now add HCl to the liquid, filter if necessary from AgCl, and pass H_2S into the acid solution; orange-red Sb_2S_3 will be precipitated.

331. **A Stain of Sb on Porcelain**, which has been produced in the manner already described in paragraph 316, differs from the As-stain by presenting a *dead* surface and by being insoluble in solution of bleaching-powder. Also when the stain is dissolved by warming it with Am_2S, and the solution is evaporated by a gentle heat, a residue of *orange-red* Sb_2S_3 is left.

332. **The Sb-mirror**, obtained as is described in paragraph 317, differs from the As-mirror by being formed much nearer to the flame and on both sides of the heated part of the tube. This proves that Sb is less volatile than As. The mirror may further be distinguished by the test which is described in paragraph 331.

The sublimate of Sb_2O_3, which is produced by heating this mirror in the air, differs from the corresponding arsenic sublimate (317) by being insoluble in boiling water. The sublimate may, however, be dissolved in a little boiling HCl. This acid solution yields an *orange-red* precipitate (Sb_2S_3) when H_2S is passed into it; whereas the solution of the As_2O_3 sublimate yields *yellow* As_2S_3 under similar conditions.

333. **$HgCl_2$.**—If SbH_3 is allowed to act upon filter-paper moistened with $HgCl_2$ solution, as is described in paragraph 318, it produces only a *greyish-brown* stain, and this is not a characteristic result.

334. **Zn and KHO**—If a solution containing Sb is boiled with Zn and KHO, it does not evolve SbH_3; hence the escaping hydrogen does not stain paper moistened with $AgNO_3$ solution (319). This test serves to distinguish As from Sb in its compounds.

Tests for Solid Compounds of Sb.

335. **Blowpipe-test on Charcoal.**—Mix a little solid substance containing Sb in a cavity on wood-charcoal with Na_2CO_3 and KCy. Heat the mixture in the inner blowpipe-flame. A white incrustation will be formed on the charcoal; and white globules of metallic Sb will be obtained, which are extremely brittle (288). The flame will also be coloured pale blue, unless this tint is concealed by the yellow sodium coloration.

When the melted Sb globules are withdrawn from the flame, they will emit a white smoke and become coated with sharp white crystals of Sb_2O_3.

For the Distinctive Tests for Antimonious and Antimonic compounds (336, 337) use $SbCl_3$ solution, and solution of potassium antimonate in water respectively.

Reagent.	**Antimon*ious*.**	**Antimon*ic*.**
336. Add excess of KHO, then $AgNO_3$ solution:	A dark-coloured precipitate which when shaken after addition of AmHO leaves black Ag_4O undissolved.	A brown precipitate, which dissolves entirely on addition of AmHO.
337. Add excess of HCl and warm; then pour in a little KI solution:	No iodine is set free; this is proved by the liquid remaining colourless, even after it has been cooled and mixed with freshly-prepared starch solution.	Iodine is liberated, giving a brown colour to the liquid and yielding a deep blue colour when starch solution is added to the cold liquid.

TIN (Sn)—Stannosum and Stannicum.

Use Stannous chloride, $SnCl_2.2H_2O$, and Stannic chloride, $SnCl_4$, dissolving each of these salts in dilute HCl.

The reactions 338 and 339 give ultimately the same results with both stann*ous* and stann*ic* compounds. Reactions 340, 341, and 342 serve to distinguish stannous from stannic salts, and to detect stannous salts in the presence of stannic salts.

338. **Zinc**, when it is immersed in the solution of a tin-salt which has been acidified with HCl, becomes coated with a spongy mass of Sn. Sn is often precipitated in thi reaction in beautiful crystals.

The Zn should be allowed to remain for some time in the solution, which is contained in a small porcelain dish; the reaction is hastened by the application of a gentle heat. The deposition of Sn from $SnCl_2$ takes place much more rapidly than from $SnCl_4$.

If the Zn rests upon platinum-foil in the solution, the Sn will be deposited in a spongy state *on the Zn and will not stain the platinum black*. Sn differs in this way from Sb.

In general analysis the deposit on the Zn may consist of other substances besides Sn. It should, therefore, be removed by rubbing and washing the Zn in some water in the porcelain dish; and the deposit may then be rinsed into a narrow test-tube, and dissolved in a little boiling strong HCl. This solution will contain $SnCl_2$, which will yield with $HgCl_2$ solution a white precipitate of Hg_2Cl_2 (280, 341).

Since the precipitation of Hg_2Cl_2 depends upon the formation of $SnCl_2$ and its presence in the acid solution, and since $SnCl_2$ is readily converted in the acid liquid into $SnCl_4$ by oxidation, precautions must be taken against exposing the acid solution freely to the air. It will usually suffice to dissolve the deposit quickly in a *narrow* test-tube, and to proceed *at once* to add the $HgCl_2$.

339. **Blowpipe-test on Charcoal.**—Mix any solid compound, containing Sn, with powdered Na_2CO_3 and KCy, and

heat the mixture on charcoal in the inner blowpipe-flame. A slight white incrustation will be obtained, together with white particles of metallic Sn which are with difficulty fused into globules.

The globules of Sn are malleable; they differ from those of Pb by their greater hardness, which prevents them from marking paper. Refer to paragraph **288** for a description of the methods of testing these globules.

DISTINCTIVE REACTIONS FOR STANN*OUS* AND STANN*IC* COMPOUNDS.

Test.	Stann*ous*. Use $SnCl_2.2H_2O$ dissolved in dilute HCl.	Stann*ic*. Use $SnCl_4$ in dilute HCl.
340. Pass H_2S:	*Dark brown precipitate* * (SnS): soluble in KHO and in *yellow* Am_2S on heating; re-precipitated by HCl, from the KHO solution as brown SnS, from the Am_2S solution as yellow SnS_2. SnS is insoluble in *colourless* Am_2S.	*Yellow precipitate* * (SnS_2), often forming only when the liquid is heated: soluble in Am_2S (both *yellow* and *colourless*) and in KHO on heating, re-precipitated by HCl as yellow SnS_2 from both solutions.
341. $HgCl_2$:	*White precipitate* (Hg_2Cl_2): turns grey on being boiled if the $SnCl_2$ is in excess (280)	No precipitate.
342 $AuCl_3$: added after a drop of Br-water:	*Purple* or *dark brown precipitate* of "Purple of Cassius."	No precipitate.

Both SnS and SnS_2 are readily soluble in boiling strong HCl.

DETECTION OF ONE METAL IN GROUP II.B., WHEN IT OCCURS ALONE.

343. **The Tests in the following Table of Differences** answer for these metals in both their sets of compounds: for distinctive tests see the preceding reactions (323-326: 336, 337: 340-342).

No. 3 may be omitted, unless decisive results have not been obtained by 1 and 2. No 4 is for solids only.

GROUP II.B.—TABLE OF DIFFERENCES.

Tests.	As.	Sb.	Sn.
For liquids.			
1. Pass H_2S into the solution acidified with HCl and heat; if no precipitate forms heat to boiling, and again pass H_2S	*Yellow* precipitate, insoluble in boiling strong HCl. Arsen*ic* compounds give this precipitate only on boiling: arseni*ous* in the cold.	*Orange red* precipitate, soluble in boiling strong HCl.	*Brown* precipitate with stann*ous*: *Yellow* precipitate with stann*ic*· Both soluble in boiling strong HCl.
2. HCl and Zn on platinum:	AsH_3 evolved, *no stain on the platinum.*	*Black stain of Sb on the platinum.*	Sn deposited on the zinc, *no stain on the platinum.*
3. Zn and HCl:	*AsH_3* gas is evolved, which if passed into $AgNO_3$ solution gives a black precipitate of Ag, H_3AsO_3 being left in solution; on adding *dilute* AmHO to this solution, yellow Ag_3AsO_3 is precipitated.	*SbH_3* gas is evolved, which if passed into $AgNO_3$ solution gives a precipitate of Ag_3Sb, from which hot $H_2\bar{T}$ solution dissolves Sb; H_2S gives in this solution after adding HCl orange-red Sb_2S_3.	*Sn* is deposited on the Zn strips: and if dissolved in boiling HCl gives a white precipitate on addition of $HgCl_2$.
For solids.			
4. Heat with Na_2CO_3 and KCy on charcoal in the inner blowpipe flame.	*No* metallic globules; smell of garlic. Confirm by heating same mixture in an ignition tube (330).	*Brittle* metallic globules, white incrustation.	*Malleable* metallic globules and slight white incrustation.

SEPARATION AND DETECTION OF METALS IN GROUP II.B., WHEN THEY OCCUR TOGETHER.

344.* **The following Differences** are utilised :—

1. The solubility of Sb_2S_3, SnS and SnS_2 in hot strong HCl, in which As_2S_3 is insoluble.

2. The difference in the reaction of Zn and HCl upon the solutions, and of SbH_3 and AsH_3 upon silver nitrate solution (315, 330).

3. The difference in the products of fusion with Na_2CO_3 and KNO_3 (347).

4. The action of Zn and Pt in the presence of HCl upon the solution, Sb staining the platinum and Sn being deposited upon the Zn (329, 338).

For the Separation and Detection of As, Sb and Sn, when they are mixed, several methods are in use. The most trustworthy are those introduced by *Hofmann* (346) and by *Fresenius* (347). The method described in par. 345, which is more simple and can be much more rapidly carried out, may, however, be generally employed.

FIRST METHOD.

345.* **From a Solution which may contain As, Sb, and Sn,** these metals are first precipitated as sulphides by passing H_2S into the acidified solution. The sulphides are then filtered off, and the filtrate is again tested by passing H_2S through it and warming, in order to see whether the precipitation has been complete. When no further precipitate is produced by H_2S the precipitated sulphides are examined in the following manner, the procedure being based on differences 1 and 4 (344).

The precipitate produced by H_2S is filtered off, and is then allowed to drain for some time upon the filter in the funnel, It is best to drain it still further; this may be effected by the filter-pump, or by opening out the filter, and spreading it upon a pile of three or four cut filter-papers. The precipitate is then removed to a small porcelain dish, and is heated for some time, nearly to boiling, with strong fuming HCl. The liquid is then cooled and filtered:—

The Residue will consist chiefly of As_2S_3, which is almost insoluble in strong HCl.

Dry the washed residue on the filter at a gentle heat, then mix it with three or four times as much powdered KCy and Na_2CO_3, and heat the mixture in a small bulb-tube, removing any drops of water inside the tube by a twisted piece of filter-paper; a black mirror (320):—**Presence of As.**

Note.—The As_2S_3 may also be dissolved by heating with a little fuming HNO_3. The excess of HNO_3 is boiled off and As detected as H_3AsO_4 by adding AmCl, excess of AmHO and $MgSO_4$ (326); or by adding $AgNO_3$ and then cautiously neutralising with AmHO (323).

The Filtrate may be examined for Sb and Sn by either I. or II. below:—

I. Place a piece of platinum-foil in a porcelain dish and pour the acid filtrate upon it, and touch the foil with a piece of Zn; H will come off with effervescence, and if either at once or after a few minutes a black stain appears upon the platinum the *presence of Sb* is indicated.*

The platinum-foil is removed and pieces of Zn are placed in the liquid; as soon as the bubbles of H cease to be given off, Zn still remaining undissolved, remove the pieces of Zn, rubbing and rinsing any dark deposit back into the dish; let this deposit settle, decant the liquid and heat the solid deposit with strong HCl for several minutes in a test-tube, dilute with a little water, filter if necessary, and add a few drops of $HgCl_2$ solution; a white or grey precipitate (280 341) indicates:—**Presence of Sn.**

II. The acid filtrate is poured into a little hydrogen flask (fig. 71, p. 173), in which H has been coming off briskly for about five minutes, being produced by the action of a little strong HCl upon some pieces of Zn. The H is lighted at the jet, and the inside of a small porcelain dish or crucible lid is pressed down upon the flame; a black stain, which is not dissolved by solution of bleaching-powder, shows:—**Presence of Sb.**

The residue in the flask is tested for Sn, as is directed in the latter part of I. (above).

*** The Sb thus deposited on the Pt may be rinsed and then dissolved by heating the foil in a test-tube with a little very dilute HNO_3; if this solution is cooled and diluted, and H_2S is passed through it, an orange-red precipitate will be formed, confirming the *presence of Sb*,**

SECOND METHOD.

346.* **Hofmann's Method.**—The sulphides, which have been precipitated as is directed in paragraph 345, are warmed with a little strong HCl, and a small crystal of $KClO_3$ is occasionally dropped in until solution is complete. The warming of the liquid is then continued until no smell of Cl is evolved.

This solution of the metals is then poured into a suitable flask, in which H is being generated by the action of HCl upon pure Zn (fig. 70, p. 171). Sn (Au and Pt) will be deposited upon the Zn, while As and Sb will be evolved as AsH_3 and SbH_3 gases. These two gases are passed into $AgNO_3$ solution, and yield soluble H_3AsO_3 and insoluble Ag_3Sb.

The $AgNO_3$ solution, containing the black precipitate, is filtered :—

Precipitate : wash this well upon the filter, and pour upon it some boiling H_2T solution. Boil this liquid again and pour it several times on the filter. Now add HCl and pass H_2S : a red coloration or precipitate indicates :— **Presence of Sb.**	**Filtrate** : add to this some very dilute AmOH, drop by drop, with constant mixing by stirring or shaking ; a pale yellow flocculent precipitate indicates :— **Presence of As.**

The Deposit of Sn is removed from the Zn in the flask, and is washed. It is then dissolved by boiling it with a little strong HCl in a narrow test-tube. For the examination of any residue here, refer to paragraph 1073 if Au and Pt have to be tested for. The solution in HCl is *at once* diluted and is tested for $SnCl_2$ by the addition of $HgCl_2$ solution: a white precipitate, possibly turning grey (280, 341), shows :—**Presence of Sn.**

A further test is made, if necessary, upon the original solution in order to ascertain whether the tin was present as stannous or as stannic salt (340–342).

THIRD METHOD.

347.* **This method depends upon the fact** that fusion with Na_2CO_3 and $NaNO_3$ converts As_2S_3 into soluble Na_2HAsO_4, Sb_2S_3 into insoluble $Na_2H_2Sb_2O_7$, and SnS_2 into insoluble Sn or SnO_2.

The Sulphides, which have been precipitated as is directed in paragraph 345, are dried on the filter, and the precipitate is well mixed with equal weights of powdered Na_2CO_3 and $NaNO_3$.

If the quantity of the precipitate is very small, the part of the paper which contains the precipitate is cut up into small pieces, and these are treated as is directed for the precipitate.

This mixture is dropped in small quantities at a time into some $NaNO_3$, which is fused in a small porcelain crucible over a Bunsen-flame. When the powder has all been added, the crucible is heated sufficiently to keep the mixture melted for a few minutes, and the liquid mass is then poured into a small dry porcelain dish.

As soon as the substance has cooled, cold water is poured upon it in the dish, and water is also poured upon the residue which adheres to the inside of the crucible. The solid substance is allowed to soak in both vessels for some time with occasional stirring, crushing it by pressure with a pestle if it does not easily fall to pieces; the liquid is then filtered :—

The Residue is washed on the filter with a mixture of equal measures of water and alcohol, in which $Na_2H_2Sb_2O_7$ is insoluble ; the washings are rejected. The precipitate is then rinsed into a small porcelain dish, using as little water as possible ; a few drops of strong HCl are added and the dish is heated, more HCl being added, if necessary, to make the liquid acid to litmus-paper. Whether the residue has dissolved or not, place in the liquid a piece of clean platinum-foil, and drop upon the foil a piece of pure Zn. Remove the foil after a few seconds: a black stain shows ,—**Presence of Sb.** See foot-note (345*). When the Zn ceases to be acted upon, Sn if present will remain as a spongy deposit This is to be washed by decantation, and then dissolved by boiling with HCl* in a test-tube : $HgCl_2$ is added immediately to the solution : a white precipitate (341) shows :—**Presence of Sn.**	**Filtrate**: HNO_3 is added until the solution is just acid after being boiled ; then $AgNO_3$ solution is poured in as long as it causes any precipitate, and very dilute AmHO is added little by little †; a brown precipitate, readily dissolving in excess of AmHO (323), shows : **Presence of As.**

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* An insoluble residue may contain Au, Pt. See paragraph 1073.

† Instead of mixing dilute AmHO with the liquid, AmHO may be carefully poured upon the top of the liquid ; a brown colour is then seen at the surface of contact.

REACTIONS OF THE RARER ELEMENTS IN GROUP II.

The Rarer Elements of this group are divided into two classes . those belonging to Group II.A. are Pd, Os, Rh, Ru ; those belonging to Group II.B. are Au, Pt, Mo, Se, Te, Ir.

GROUP II.A.—COPPER GROUP.

In this Group are included Pd, (Os, Rh, Ru) ; these metals are precipitated as sulphides by H_2S from acid solutions, and the sulphides are insoluble in Am_2S and in solution of alkaline hydrate.

PALLADIUM (Pd).—Use solution of Palladium chloride, $PdCl_2$.

Pd occurs as metal in native platinum, also in gold and in silver. Palladium solutions are reddish-brown, and yellow if dilute : the addition of water precipitates from these solutions a basic salt, unless sufficient free acid is present to prevent the precipitation.

348. **H_2S** : black precipitate in neutral, alkaline, and acid solutions ; this precipitate is insoluble in Am_2S, but soluble in boiling HCl and in aqua regia.

349. **AmHO** : flesh coloured precipitate ($PdCl_2.2NH_3$) ; soluble in excess of AmHO to a colourless liquid, from which HCl precipitates ellow crystalline palladammonium chloride ($N_2H_6Pd''Cl_2$).

350. **$HgCy_2$** : yellowish-white gelatinous precipitate ($PdCy_2$), slightly soluble in HCl, easily soluble in AmHO. This is a very characteristic reaction.

351. **KI** : black precipitate (PdI_2) ; a very characteristic reaction.

GROUP II.B.—ARSENIC GROUP.

In this Group are included Au, Pt, Mo, Se, Te, (Ir).

GOLD (Au).—Use solution of Gold chloride, $AuCl_3$.

The solution is light yellow in colour. It yields a dark brown sulphide, which is soluble in yellow Am_2S_n.

352. **$SnCl_2$ containing a little $SnCl_4$** : purple or dark brown precipitate of *Purple of Cassius*, which is best seen when the test is made in a white porcelain dish.

353. **$FeSO_4$ solution** produces either at once, or when the solution is heated, a very finely-divided precipitate of Au: the liquid usually appears bluish by transmitted light, and is always copper-red and turbid by reflected light:—

$$2AuCl_3 + 6FeSO_4 = 2Au + 2Fe_2(SO_4)_3 + Fe_2Cl_6.$$

354. **H_2SO_3 solution** produces a similar precipitate of Au when the liquid is boiled. If the liquid is boiled for some time in a porcelain dish, the Au settles down in small black masses,' and the solution loses its colour.

PLATINUM (Pt).—Use solution of Platinic chloride, $PtCl_4$.

The solution is reddish-yellow in colour. It yields a dark brown sulphide, which is soluble in yellow Am_2S_n

355. **AmCl**, added to a strong solution, produces on standing for some time, or more quickly on being stirred, a yellow precipitate (Am_2PtCl_6), which is soluble in much hot water.

356. **$SnCl_2$** gives a reddish-brown colour ($PtCl_2$) in the solution acidified with HCl:—$PtCl_4 + SnCl_2 = PtCl_2 + SnCl_4$.

The Method of Separating and Detecting Au and Pt is fully given in paragraph 1073.

MOLYBDATE (MoO_4)—Use Am_2MoO_4 solution.

Mo occurs as molybdate: also as sulphide, which may be readily converted into MoO_3 by heating it in the air or with HNO_3. Unignited MoO_3 dissolves in acids; ignited MoO_3 is insoluble in acids, but is easily soluble in alkalies.

357. **HCl, HNO_3, or H_2SO_4**, if it is added in small quantity to an aqueous solution of a molybdate, yields a precipitate, which is readily soluble in excess of the acid.

358. **H_2S**, added in *very small* quantity to the acidified solution, gives a *blue* liquid; if H_2S is added in larger quantity, a *brown* precipitate (MoS_3) forms; the precipitation becomes complete only when the solution is heated and H_2S is passed for some time. This precipitate is soluble in solution of alkaline sulphide or of alkaline hydrate; MoS_3 is reprecipitated from this solution when acid is added in excess.

359. **Zn, or $SnCl_2$**, if it is added to the solution of a molybdate in HCl, colours it *brown*, *green*, or *blue*, according to its state of concentration.

360. **KCyS**, if it is added to a solution acidified with HCl, gives no coloration; but if a piece of Zn is afterwards dropped into the liquid, a beautiful *crimson* colour is produced when this liquid is shaken with ether, the colour is taken up by the ether.

361. **Na_2HPO_4**. if it is added in very small quantity to the solution of molybdate acidified with HNO_3, gives a yellow precipitate when the liquid is gently warmed; this precipitate is readily soluble in excess of alkaline hydrate solution.

362. **Borax-bead**: outer flame, *yellow*; inner flame, *dark brown*.

363 **Microcosmic-bead**: outer and inner flames, *green*.

SELENIUM (Se).—Use FeSe, K_2SeO_3, and K_2SeO_4.

Se occurs as selenide of Pb, Fe, Cu, Ag.

I Selenide (Se).—Use FeSe.

364. **When a Selenide is heated** in an open tube it evolves a smell of decaying horse-radish, and produces a grey or red sublimate of Se · the smell is very characteristic.

II. Selenite (SeO_3).—Use K_2SeO_3 (369).

365. **H_2S** gives in an acid solution, if cold a *yellow*, if hot a *reddish-yellow* precipitate this precipitate is soluble in Am_2S.

366. **$BaCl_2$** gives in a neutral solution a white precipitate: soluble in HCl and in HNO_3

367. **$SnCl_2$ or H_2SO_3** gives in the presence of free HCl a red, or in warm solutions a grey precipitate of Se.

368. **Cu** in a hot solution acidified with HCl becomes covered with a black film: the liquid, on standing with the Cu for some time, becomes coloured red by Se.

III. Selenate (SeO_4).—Use K_2SeO_4.

369. **HCl** produces no change in the cold; but on boiling, Cl is given off and the selenate is reduced to selenite, to which the above tests (365–368) may be applied.

370. **$BaCl_2$**: a white precipitate ($BaSeO_4$); insoluble in cold HCl; dissolved by boiling with HCl, with evolution of Cl and reduction to $BaSeO_3$.

IV. Selenium in any form of Combination.

371 **When it is heated on Charcoal in the Inner Blowpipe-flame** a solid selenium compound evolves a smell of rotten horse-radish.

372. **When it is heated on Charcoal with Na_2CO_3 in the Inner Blowpipe-flame** a selenium compound yields a fused mass, which when cooled and moistened on Ag yields a black stain, and on addition of HCl evolves H_2Se, a badly smelling gas.

TELLURIUM (Te).—Use PbTe, K_2TeO_3, K_2TeO_4, as is directed below.

Te commonly occurs united with the metals Au, Ag, Bi, Cu, Fe.

I. Telluride (Te).—Use PbTe.

373. **When a Telluride is heated in an open glass tube** it gives white fumes and a sublimate; the sublimate differs from that given by Sb, by being fusible before the blowpipe.

II. Tellurite (TeO_3).—Use K_2TeO_3 (377).

374. **H_2O** : on dilution with water, tellurous acid is precipitated from the acid solution.

375. **H_2S**: in acid solutions a brown precipitate (TeS_2), easily soluble in Am_2S.

376. **H_2SO_3, $SnCl_2$, or Zn** precipitates black Te.

III. Tellurate (TeO_4).—Use $KeTeO_4$.

377. **HCl** produces no change in the cold solution of a tellurate; but on boiling, Cl is evolved and the tellurate is reduced to tellurite; the solution then gives the above reactions (374–376).

IV. Tellurium in any form of Combination.

378. **If it is fused with Na_2CO_3 on Charcoal in the Inner Blowpipe-flame**, a tellurium compound gives sodium telluride, a solution of which gives a black stain on Ag. When this solution is acidified it deposits black Te, and evolves H_2Te.

For the Detection of the Rarer Elements in Group II. see the second column in Table, paragraph 1090.

GROUP I.—SILVER GROUP.

379. This Group includes Pb, Ag, Hg′, together with the rarer elements Tl and W.

The solutions of these metals differ from those of all other metals, by being precipitated as chlorides by HCl or by a dissolved chloride. Since, however, $PbCl_2$ and TlCl are somewhat soluble in water, these metals are not entirely precipitated in this Group as chlorides, and they will also be found in Group II.A. as sulphides in the course of analysis.

For the Rarer Elements in this Group refer to paragraphs 391–400.

LEAD (Pb).

380. The reactions for Pb have been already given under Group II.A. in paragraphs 284–288.

SILVER (Ag).—Use $AgNO_3$ solution.

381. **HCl**: white precipitate (AgCl), which becomes curdy when it is shaken or heated.

Pour off the liquid with the precipitate into four tubes. Filter one portion, wash the precipitate and let it stand for some time exposed to sunlight or to common daylight; it will become blackish-purple.

Show with the other portions that AgCl dissolves readily when it is heated with AmHO or with KCy-solution, and is precipitated again from these solutions when HNO_3 is added in excess. Show also that AgCl is insoluble in HNO_3, even when the acid is boiled.

382. **H_2S or Am_2S:** black precipitate (Ag_2S), insoluble in Am_2S and in KHO, soluble in boiling dilute HNO_3.

383. **KHO:** brown precipitate (AgHO), insoluble in excess.

384 **AmHO:** light brown precipitate, produced only when *very dilute* AmHO is added drop by drop, since AgHO is very easily soluble in excess of AmHO.

385. **Blowpipe-test on Charcoal.**—If any solid substance containing Ag is mixed with powdered Na_2CO_3 and the mixture is then heated on charcoal in the inner blowpipe-flame, white malleable globules or scales of Ag will be obtained, but no incrustation will be formed on the charcoal.

MERCUROSUM (Hg′).—Use Mercurous nitrate, $Hg'_2(NO_3)_2.H_2O$, dissolved in dilute HNO_3.

Reactions 279–283 for mercuric salts yield precisely similar results with mercurous salts The two following reactions (386, 387), however, serve to distinguish mercuric from mercurous salts.

386. **HCl:** a white precipitate (Hg'_2Cl_2), insoluble in cold dilute acids, but soluble when it is warmed with HNO_3 or with a mixture of HCl and HNO_3, which convert it into soluble mercuric salt.

This precipitate *becomes black when AmHO is poured upon it, but does not dissolve.*

A solution of mercur*ic* salt is not precipitated by HCl or by solution of a chloride.

387. **AmHO, or KHO:** *black* precipitate, insoluble in excess.

In a solution of mercur*ic* salt AmHO usually causes a *white* precipitate (NH_2HgCl), and KHO precipitates *yellow* HgO.

DETECTION OF ONE METAL IN GROUP I., WHEN IT OCCURS ALONE.

388. The following Table of Differences enables one metal of this Group to be detected, when it is present alone.

GROUP I.—TABLE OF DIFFERENCES.

Tests.	Pb.	Ag	Hg'.
For liquids.			
1. Add HCl:	White precipitate, *soluble in boiling water; AmHO neither dissolves the precipitate nor changes its colour.*	White curdy precipitate, *insoluble in boiling water, easily soluble in warm AmHO.*	White precipitate, *insoluble in boiling water, insoluble in AmHO, but blackened by it.*
2. Add AmHO, very dilute.	*White precipitate,* insoluble in excess.	*Brown precipitate,* readily soluble in excess.	*Black precipitate,* insoluble in excess.
3. Add K_2CrO_4:	*Bright yellow* precipitate.	*Chocolate red* precipitate.	*Scarlet red* precipitate.
For solids.			
4. Fuse with Na_2CO_3 on charcoal in the inner blowpipe flame.	*White malleable globules of metal* which mark paper, yellow incrustation on the charcoal	*White malleable globules or scales* which do not mark paper; no incrustation	No metallic globules Confirm by heating with Na_2CO_3 in an ignition tube (282).

SEPARATION AND DETECTION OF METALS IN GROUP I., WHEN THEY OCCUR TOGETHER.

389.* The following Differences are made use of :—

1. The solubility of $PbCl_2$ in boiling water, in which $AgCl$ and Hg'_2Cl_2 are insoluble.

2. The solubility of AgCl in AmHO, in which Hg'_2Cl_2 is insoluble.

After each metal has been separated, its presence is confirmed by one of its characteristic tests.

390.* A Solution which is to be examined for Pb, Ag, and Hg′ is precipitated by adding HCl in excess; it is then filtered, and the clear filtrate is mixed with more HCl in order to ascertain whether the metals have been completely precipitated. The precipitate is then examined by Table I. (**1022**).

REACTIONS FOR THE RARER ELEMENTS IN GROUP I.

391. The elements included in this Group are Tl and W.

The addition of HCl in excess partially precipitates Tl as chloride, and completely precipitates tungstic acid from a tungstate

Since TlCl is only partially precipitated in Group I., owing to its being somewhat soluble in water, Tl is also found in the ordinary course of analysis in Group III B., where it is completely precipitated as sulphide by Am_2S.

THALLIUM (Tl).—Use Tl_2SO_4 solution.

Tl occurs in small quantity in many natural sulphides, and occasionally in the ashes of plants and in mineral waters. Tl yields both thall*ic* and thall*ious* salts; but the former are very unstable, changing, even when their solutions are heated, into thall*ious* salts.

392. **HCl**. a white precipitate, which rapidly settles, does not blacken in the light, and is soluble in aqua regia. It is soluble in a large quantity of water, and therefore does not form in dilute solutions.

393. **KI** a yellow precipitate, almost insoluble in water, more soluble in KI solution. If ferric salt is present in the solution, it must be reduced by H_2SO_3 before KI is added.

394 **$PtCl_4$** : orange-red precipitate, slightly soluble in water.

395. **Am_2S** black precipitate, which is easily coagulated by heat is insoluble in AmHO, in alkaline sulphides, and in KCy-solution; is readily oxidised by the air to Tl_2SO_4, and is easily soluble in mineral acids

Tl is completely precipitated by H_2S from a solution in which $H\bar{A}$ is the only free acid present, but the presence of a free mineral acid prevents the precipitation.

396. **Flame-Coloration.**—Thallium compounds impart to the Bunsen-flame an intense *green* colour, which rapidly disappears.

The spectrum (fig. 68, p 100) consists of one bright emerald-green line, and is very characteristic. Thallium may be easily detected by the spectroscope in solution, or in any of the precipitates which are mentioned above.

TUNGSTATE (WO_4).—Use Na_2WO_4 solution.

W usually occurs in the form of a tungstate. An insoluble tungstate yields a soluble alkaline tungstate when it is fused with an alkaline carbonate. From the solution of an alkaline tungstate HCl precipitates the tungstic acid entirely.

397. **HCl, HNO_3, or H_2SO_4**: white precipitate (H_2WO_4), which becomes yellow when the liquid is boiled it is insoluble in excess of acid, but soluble in AmHO.

If a piece of Zn is dropped into the acid liquid containing the precipitate, it yields a deep blue colour.

398. **$SnCl_2$** in neutral solution, made by dissolving $SnCl_2$ crystals in water and filtering, gives a *yellow* precipitate, which becomes *blue* when HCl is added and the liquid is heated.

399. **Am_2S** yields no precipitate in the solution of an alkaline tungstate; but if Am_2S is added, and the liquid is then made acid with HCl, brown WS_3 is precipitated.

400. **Microcosmic-bead**: outer flame, *colourless or yellow*. inner flame, *blue*: if a little $FeSO_4$ is introduced into the fused bead, the colour changes to *blood-red*. These colours are best seen when the bead is perfectly cold.

For the Detection of the Rarer Elements of this Group, refer to the first column in the Table (1090).

DETECTION OF METALS BY THE ANALYTICAL TABLES

401. The metals are classified in Analytical Groups in paragraph **931.**

The more important tests for the metals are brought together in systematic and tabular form in Sections V. and VI.

The scheme of analysis in Section V. serves to detect the presence of one metal when all other metals are absent. This Section is only employed for special purposes.

The fuller scheme of analysis in Section VI. serves to detect metals when they are present together.

The General Table for the detection of the Rarer Elements will be found in paragraph **1090.** This is supplementary to the General Table (**1007**).

SECTION IV.—PART III.

ANALYTICAL REACTIONS FOR ACID-RADICLES.

498. **Introductory Remarks.**—The Acid-radicles are usually detected in the course of analysis by special tests. They cannot advantageously be precipitated in Groups, from which the members of each Group are subsequently separated and identified, as is the case with the Metals.

Accordingly the classification adopted for their reactions consists in placing together in a Group those Acid-radicles which somewhat resemble one another in their reactions; and then stating at the end of each Group upon what differences the detection of its members, when they occur together, depends.

The reactions for Acid-radicles are worked through in the same way as those for the Metals, a salt or other compound which contains the radicle being employed. At the end of each Group the student may with advantage detect one or more of its members, as was done with the Metals.

The systematic Tables of Differences, such as are given for each Group of the Metals, are not drawn out for the Acid-radicle Groups. The student should have no difficulty in constructing such Tables for himself, either mentally or on paper, after looking through the reactions

GROUP I.—SULPHATE GROUP.

499. This Group includes sulphate (SO_4), fluosilicate (SiF_6), and selenate (SeO_4).

These acid-radicles are distinguished by yielding a precipitate, when $BaCl_2$ is added in the presence of hydrochloric acid.

Sulphate is the only commonly occurring acid-radicle which gives with $BaCl_2$ a precipitate insoluble in boiling HCl

Fluosilicate resembles sulphate in this respect, but differs from sulphate in so many reactions that it is considered hereafter (610–614) in connection with fluoride and silicate, to which it is analytically more closely related.

Selenate yields a precipitate of $BaSeO_4$ when $BaCl_2$ is added in the presence of cold HCl; but this precipitate differs from $BaSO_4$ by being dissolved when it is boiled for some time with strong HCl (370)

SULPHATE (SO_4).—Use $Na_2SO_4.10H_2O$.

500. $BaCl_2$, or $Ba(NO_3)_2$; white precipitate ($BaSO_4$); remains undissolved when it is boiled with dilute HCl or HNO_3.

Note.—$BaCl_2$ and $Ba(NO_3)_2$ are insoluble in strong HCl and in strong HNO_3. Care must be taken not to mistake the precipitates which are caused by these acids for a precipitate of $BaSO_4$. These precipitates are readily distinguished from $BaSO_4$ by their solubility in water.

Add $BaCl_2$ or $Ba(NO_3)_2$ solution to strong HCl or HNO_3. A white precipitate will be produced, consisting of $BaCl_2$ or of $Ba(NO_3)_2$, which is insoluble in the strong acid. Now add much water: the precipitate will dissolve readily and completely in the water.

501. Blowpipe-test on Charcoal.—If a sulphate, or any solid substance containing sulphur, is mixed with solid Na_2CO_3, and the mixture is then heated on charcoal in the inner blowpipe-flame until the Na_2CO_3 fuses, Na_2S will be produced:—

$$Na_2SO_4 + 4C = Na_2S + 4CO.$$

The Na_2S thus produced may be readily detected by detaching the cold solid mass from the charcoal with the point of a knife, placing a portion of it upon a bright silver coin, and moistening it with a drop of water. After the moistened mass has remained upon the coin for a short time, it is rinsed off; a black stain (Ag_2S) will then be seen upon the part of the silver upon which the substance rested (**511**).

Another portion of the cool mass may be moistened with a drop of HCl. A fetid smell of H_2S will then be observed; and a piece of paper, moistened with $Pb\bar{A}_2$-solution and held above the substance, will be blackened (**510**).

This test is evidently only of value for detecting a sulphate, when it is known that sulphur is not present in any other form in the substance to be tested

It is also necessary that sulphur should not be introduced by the Na_2CO_3, or by the flame. Since coal-gas may contain sufficient sulphur to give the above reaction with pure Na_2CO_3, it is advisable to employ the flame of a spirit- or petroleum-lamp (3) rather than that of a gas-burner for this reaction.

502. **Hydrogen sulphate, or Sulphuric acid (H_2SO_4),** even when it is dilute, may be detected by producing black charred stains on paper, which has been dipped into the liquid and has then been dried by heat. Sulphuric acid has a strongly acid reaction to litmus, and evolves hydrogen when it is diluted and warmed with Zn: these properties are, however, shared by some metallic sulphates and by the acid sulphates.

SELENATE (SeO_4) :—The reactions for selenate have been already given (369–372) in Group II. for the metals. Selenate is classed in Group II. because it gives a precipitate with H_2S, after its solution in hydrochloric acid has been boiled.

FLUOSILICATE (SiF_6) see reactions 610–614.

DETECTION OF ACID-RADICLES OF GROUP I.

503. After the acid-radicle has been precipitated by the addition of $BaCl_2$, it may be identified by noting the following differences in the properties of the insoluble barium salts.

The precipitate of $BaSO_4$ differs from that of $BaSeO_4$ by being quite unaffected when it is boiled with HCl.

$BaSeO_4$ may be dissolved by being boiled with HCl, and selenite may then be detected in the solution by the tests given in paragraphs **365–368**. When $BaSeO_4$ is heated on charcoal, it gives the peculiar smell of rotten horse-radish.

$BaSiF_6$ is distinguished by heating it with strong H_2SO_4, when HF is evolved, which etches glass (**612**). For other differences, see paragraphs **610, 611**.

GROUP II.—CARBONATE GROUP.

504. This Group includes carbonate (CO_3), sulphide (S), sulphite (SO_3), thiosulphate (S_2O_3), hypochlorite (ClO), and nitrite (NO_2).

Salts which contain these acid-radicles evolve characteristic gases when they are acted upon by HCl.

CARBONATE (CO_3).—Use Na_2CO_3, or $CaCO_3$.

505. HCl, $\bar{A}$, $H_2\bar{T}$, and almost any other acid, when it is poured upon a carbonate in a test-tube, causes colourless, almost inodorous carbon dioxide gas (CO_2) to escape with effervescence. This gas is recognised by its property of turning lime-water or baryta-water milky, owing to the production of an insoluble carbonate.

The Lime-water Test may be tried in the several ways described below, attention being paid to the precautions on page 202.

1. A glass rod which has been just removed from some perfectly clear lime-water, and which has a small drop adhering to its end, is introduced into the test-tube in which the carbon dioxide has been evolved ; the drop will quickly become milky. Care must be taken not to allow the rod to touch the liquid or the inside of the tube The milkiness is best seen when the rod is removed and the drop is held above a dead black surface.

2. Since carbon dioxide gas is much heavier than air, it will remain in the test-tube in which it has been liberated, if the tube is held erect and its mouth is loosely closed with the thumb. If the tube is then gradually inclined, the heavy gas may be poured into another test-tube containing lime-water, without allowing any liquid to flow out. On closing this tube with the thumb, and shaking the lime-water up with the gas, the liquid will become milky.

3. The acid may be poured upon the carbonate in a small beaker, which is immediately covered with a watch-glass the under surface of which has a drop of clear lime-water adhering to the middle . the drop will become milky.

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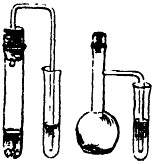
2. Since carbon dioxide gas is much heavier than air, it will remain in the test-tube in which it has been liberated, if the tube is held erect and its mouth is loosely closed with the thumb. If the tube is then gradually inclined, the heavy gas may be poured into another test-tube containing lime-water, without allowing any liquid to flow out. On closing this tube with the thumb, and shaking the lime-water up with the gas, the liquid will become milky.

3. The acid may be poured upon the carbonate in a small beaker, which is immediately covered with a watch-glass the under surface of which has a drop of clear lime-water adhering to the middle . the drop will become milky.

4. If the CO_2 is given off in considerable quantity, it may be conducted into some lime-water contained in a separate tube. This is effected by either of the forms of apparatus which are shown in figure 73

Precautions.—In testing for carbonate the following precautions must be attended to.

FIG. 73.



APPARATUS FOR TESTING GAS EVOLVED.

The milkiness, which is at first produced in lime-water by CO_2, gradually disappears when more CO_2 is absorbed. Hence, if the lime-water does not become milky *at once*, it must be constantly watched, in order to avoid the risk of the milkiness not being observed before it has been removed by the further absorption of CO_2

If the addition of the first few drops of acid to the substance does not cause an effervescence of CO_2 more acid should be added, since a carbonate may be simply converted into an acid carbonate by the action of a small quantity of acid, and it will evolve no CO_2 during this change

A powdered solid substance, which is being tested for a carbonate by HCl, should be first moistened in order to drive out the air-bubbles entangled within it, since if these escaped on the addition of the acid they might be mistaken for a slight effervescence of CO_2

HYDRIC CARBONATE, OR BICARBONATE.

506 **Hydric carbonate, or Bicarbonate,** is changed by heat into normal carbonate with evolution of CO_2 and H_2O:—$2NaHCO_3 = Na_2CO_3 + CO_2 + H_2O$. Prove this by heating some sodium bicarbonate ($NaHCO_3$) in an ignition-tube, drops of water will condense and CO_2 may be detected by lime-water (505). Also drop some $NaHCO_3$ into boiling water in a test-tube; effervescence will take place, and the escaping gas will give rise to milkiness in lime-water.

507. **$MgSO_4$.**—A solution of bicarbonate, which has been made with *cold* water, gives no precipitate with $MgSO_4$-solution; but after the solution has been boiled it will contain a normal carbonate, and this will give a precipitate of white $MgCO_3$.

508. **$HgCl_2$** yields with solution of bicarbonate a *pale yellow* precipitate; solution of *normal carbonate* yields an *orange-red* precipitate with $HgCl_2$ solution.

509. **Hydrogen carbonate, or Carbonic acid** (H_2CO_3), can only exist in dilute aqueous solution. The addition of sufficient lime-water

to its solution causes a permanent milkiness. Other substances in solution, however, behave in a similar way; hence the acid is best found by boiling the liquid, and passing the mixture of steam and CO_2 which is evolved into lime-water; the lime-water will become milky. Bicarbonates give the same result (506).

SULPHIDE (S).—Use solid FeS, or solution of Am_2S.

510. **HCl, or H_2SO_4,** when it is poured upon many sulphides, produces H_2S, which may escape with effervescence or may in great part remain in solution until the liquid is heated. This gas is detected by its fetid smell, which resembles that of rotten eggs; and by its property of darkening $Pb\bar{A}_2$-solution, owing to the formation of PbS.

The gas may be made to act upon the $Pb\bar{A}_2$-solution by the methods given for CO_2 (505, 1–4); or a small strip of filter-paper, moistened with the solution, may be introduced into the gas on the end of a glass rod. The paper strip should be attached to the rod by only half its length, so as to expose both surfaces of the lower part of the strip to the action of the gas.

Note.—This test is made much more delicate, if the filter-paper is moistened with a solution, which is prepared by adding KHO to boiling $Pb\bar{A}_2$-solution, until the precipitate, which is formed at first, is just redissolved. The test-paper, which has been thus prepared, becomes intensely blackened by H_2S.

511. **Ag.**—If solution of sulphide is dropped upon a bright silver coin, it produces a black stain (Ag_2S). This stain may be removed by rubbing the coin with moist lime.

A solid sulphide, which gives off H_2S on the addition of HCl, produces a black stain when it is placed upon a silver coin and is moistened with a drop of HCl.

512. **Solution of $Pb\bar{A}_2$** gives with solution of sulphide a black precipitate (PbS). The solution produced by adding KHO in excess to $Pb\bar{A}_2$-solution (510, *Note*) is a more delicate reagent for this test.

513. **The formation of Sulphocyanide** (650) may be

employed as a very delicate test for a soluble sulphide. It depends on the detection, by means of Fe_2Cl_6, of the sulphocyanide which has been formed by the action of KCy-solution upon the sulphide.

514. **Ignition**.—Free sulphur and many sulphides, when they are strongly heated in a test-tube, give a sublimate of sulphur in the form of brown drops or of yellow powder.

515. **Ignition in the Air.**—Free sulphur and many sulphides, when they are heated in a tube open at both ends and held obliquely in the flame, produce SO_2 gas. This gas may be recognised by its pungent smell, and by causing the colour of paper moistened with $K_2Cr_2O_7$-solution to change to bluish-green.

516. **Insoluble Sulphide, when fused with Na_2CO_3** in a covered porcelain crucible or in a hard glass ignition-tube, yields soluble Na_2S, which may be detected by the above tests (510-513).

517. **Fusion with KNO_3**, or simply heating with strong HNO_3, converts a sulphide into a sulphate, which may be detected in solution by $BaCl_2$ (500).

518. **Hydrogen sulphide, or Hydrosulphuric acid (H_2S),** in aqueous solution, is easily recognised by the tests described in paragraphs 511, 512. Unless the solution is very dilute it will emit H_2S, which may be readily detected either by its smell, or by suspending lead acetate paper over the liquid (510).

SULPHITE (SO_3).—Use $NaHSO_3$.

519. **HCl, or H_2SO_4**, when it is poured upon a sulphite and warmed, evolves SO_2 gas: this gas may be recognised by its pungent smell and by turning $K_2Cr_2O_7$-solution green.

A drop of the $K_2Cr_2O_7$-solution, hanging on the end of a glass rod, may be exposed to the gas (505, 1); or a small strip of filter-paper may be wetted with the bichromate solution and then attached by half its length to the rod, the other half hanging freely in the gas.

520. **$BaCl_2$**. white precipitate ($BaSO_3$). The formation of this precipitate is prevented by the presence of HCl, but a precipitate usually forms owing to the presence of some sulphate in the salt used When Cl-water or Br-water is added to the clear solution of $BaSO_3$ in HCl, $BaSO_4$ is formed and precipitated —

$$BaCl_2 + H_2SO_3 + H_2O + 2Cl = BaSO_4 + 4HCl.$$

521. **Nascent Hydrogen.**—The addition of sulphite solution to a mixture of HCl and Zn, which is giving off pure H, immediately causes an evolution of H_2S. The H_2S is detected by its smell, or by blackening a piece of filter-paper moistened with solution of $Pb\bar{A}_2$, or of PbO in excess of KHO (510, *Note*).

522. **Hydrogen sulphite, or Sulphurous acid** (H_2SO_3) can be detected in aqueous solution by adding to the liquid a drop of $K_2Cr_2O_7$-solution : the reddish colour will at once change to green. Strong aqueous solution of hydrogen sulphite slowly evolves SO_2, and dilute solutions evolve SO_2 when they are heated . this gas may be readily detected by its smell and by $K_2Cr_2O_7$-solution (519).

THIOSULPHATE, OR HYPOSULPHITE (S_2O_3).

Use solution of $Na_2S_2O_3.5H_2O$.

523. **HCl, or H_2SO_4**, causes no immediate change in the *cold* solution ; but after a few seconds the acidified liquid becomes turbid, owing to the separation of finely-divided yellow S, and it then contains H_2SO_3 :—$H_2S_2O_3 = S + H_2SO_3$. SO_2 from the H_2SO_3 may be recognised by its pungent smell, when the acidified liquid is heated ; and it may be detected in the solution, by adding a few drops of $K_2Cr_2O_7$-solution, which changes colour to green.

524. **Fe_2Cl_6** gives a reddish-violet colour with solution of thiosulphate. This colour disappears after a short time in the cold, and immediately when the liquid is heated. The Fe_2Cl_6-solution at the same time loses its yellow colour, since it is changed from Fe_2Cl_6 into $FeCl_2$

525. **$AgNO_3$** a white precipitate ($Ag_2S_2O_3$), which becomes black after standing for a short time, or immediately when the liquid is heated, owing to the formation of Ag_2S —

$$Ag_2S_2O_3 + H_2O = Ag_2S + H_2SO_4$$

The above precipitate ($Ag_2S_2O_3$) dissolves very readily in excess of the $Na_2S_2O_3$-solution, hence it is most easily obtained by dropping the thiosulphate solution into some $AgNO_3$-solution.

526. **Hydrogen thiosulphate, or Thiosulphuric acid ($H_2S_2O_3$)**, is extremely unstable, rapidly separating into $S + H_2SO_3$ (523).

HYPOCHLORITE (ClO).—Use solution of Bleaching-powder, containing $CaCl_2O_2$.

527. **HClO is evolved from a hypochlorite** when it is in the solid state or in strong solution; it is probably liberated by the action of the atmospheric carbon dioxide. The smell of the vapour thus evolved resembles that of Cl, but is far less irritant to the nose

528. **HCl, or H_2SO_4**, if it is added in excess, sets free Cl from hypochlorite in the cold. This gas may be recognised by its smell, by its yellowish colour, and by its power of bleaching a piece of moistened litmus-paper when the paper is held for a short time in the gas. If the Cl is small in quantity and remains dissolved, a piece of litmus-paper may be shaken up with the solution.

$$CaCl_2O_2 + 4HCl = 4Cl + CaCl_2 + 2H_2O.$$

The bleaching of litmus by solution of hypochlorite occurs slowly even without the addition of acid, owing to the bleaching power of the nascent oxygen liberated from the hypochlorite. When the hypochlorite solution is heated, or the paper moistened with the solution is exposed to the CO_2 of the air, the bleaching is hastened.

On the addition of an acid, however, the bleaching of the litmus-paper immersed in the solution is extremely rapid.

529 **$Pb\bar{A}_2$.** If solution of hypochlorite is added in large quantity to $Pb\bar{A}_2$-solution, it gives a white precipitate; this precipitate becomes red and then *dark brown* (PbO_2) when the liquid is boiled for a short time.

When hypochlorite is heated with $MnCl_2$-solution, it also gives a *dark brown* precipitate (MnO_2).

530. **Hydrogen hypochlorite, or Hypochlorous acid ($HClO$),** is a yellow liquid with sweetish smell, it is very unstable, unless it is largely diluted This acid bleaches litmus, and evolves Cl when it is warmed with HCl.

NITRITE (NO_2).—Use KNO_2.

531. **H_2SO_4,** when it is warmed with nitrite, causes a reddish-brown gas with a characteristic smell to be evolved. The coloured gas is usually best seen by looking down the test-tube at a white surface.

532. **Cold $FeSO_4$-solution,** when it is added to solution of nitrite, causes the liquid to assume a dark colour. The colour becomes much more intense when cold dilute H_2SO_4 is added. The dark coloured substance which is produced is a compound of NO with $FeSO_4$; this compound is decomposed when the liquid is boiled, and reddish-brown gas is evolved (531).

533. **Nitrous acid liberates Iodine from Potassium iodide.**—The nitrous acid may be formed by the addition of acid to a nitrite.

Drop *a little* starch-powder into some cold water in a porcelain dish, and stir and boil the liquid for a short time: then cool the starch-solution, and add to it a few drops of KI-solution.

Add to this liquid some solution of nitrite, and then several drops of $H\bar{A}$. An intense blue colour will be produced. This is due to the combination of the starch with the iodine, which has been set free by the HNO_2.

The liquid will often appear black at first, and the blue colour will then only become visible when much water has been added.

This is a most delicate test if the liquid is quite cold. But the presence of a nitrite is not proved if the colour is produced before the $H\bar{A}$ is added.

534. **Hydrogen nitrite, or Nitrous acid** (HNO_2), is extremely unstable, and decomposes rapidly into HNO_3, NO, and H_2O. It therefore evolves reddish-brown gas when its solution is heated in the air (531). Nitrous acid also colours $FeSO_4$-solution (532), and liberates iodine from KI (533).

DETECTION OF THE ACID-RADICLES IN GROUP II.

535. **The Detection of these Acid-radicles**, when they occur singly, presents no difficulty.

Their detection, when they occur together, is often complicated, since most of the acids, which are formed on the addition of HCl to salts containing acid-radicles of this Group, react upon one another. Thus HClO oxidises HNO, H_2S, and H_2SO_3; and H_2SO_3 oxidises H_2S.

The analyst may often succeed, by a skilful use of the above reactions, in detecting these acid-radicles when they are mixed. But their detection in certain mixtures is a problem which is only soluble by careful consideration, and is not unfrequently insoluble.

SO_2 may be detected by $K_2Cr_2O_7$-paper : H_2S by $Pb\bar{A}_2$-paper: Cl by bleaching moist litmus-paper : nitrite by evolving reddish-brown gas. But CO_2 can only be detected in the presence of much SO_2 by passing the gases through hot $K_2Cr_2O_7$-solution; the SO_2 will be absorbed, and the CO_2 which passes on may then be detected by lime-water.

GROUP III.—NITRATE GROUP.

536. This Group includes nitrate and chlorate.

Salts, which contain these acid-radicles, resemble one another in many respects, more particularly in the fact that none of them are perfectly insoluble in water; hence no method of precipitating them is known

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NITRATE (NO_3).—Use KNO_3 and $Pb(NO_3)_2$.

537. $FeSO_4$-solution gives a dark brown colour with Nitric acid (532). Nitric acid may be formed from a nitrate by the addition of H_2SO_4.

Add to some solution of nitrate, contained in a rather broad test-tube, about an equal volume of strong H_2SO_4. Mix the liquids well, and then cool the mixture in a stream of cold water.

Now pour some strong cold $FeSO_4$ solution in a gentle stream down the inside of the tube, which is held in an inclined position. A distinct layer of the $FeSO_4$ solution should be formed on the surface of the acid liquid. A *dark coloured* film or ring, which sometimes extends upwards, will appear at the surface of contact of the two liquids.

The colour is most easily seen when a piece of filter-paper is held between the tube and a bright flame or window, and when one looks through the tube and paper at the light. A mere *yellow colour* is often produced in the absence of a nitrate, and may be disregarded.

The test is made more delicate by cooling the liquid, since heat prevents or removes the dark colour. For this purpose the tube may be allowed to stand in a beaker of cold water for a few minutes.

If much nitric acid is present, the dark colour extends upwards as soon as the strong sulphuric acid is mixed with the $FeSO_4$ solution; but the heat which is evolved by the dilution of the strong H_2SO_4 soon causes the NO to escape and to form a reddish-brown gas in the test-tube; the liquid at the same time loses its dark colour.

538. **Copper.**—Drop a few small scraps of Cu into some nitrate solution, and add strong H_2SO_4. Reddish-brown gas will appear either at once or when the tube is warmed. If the colour is faint, it is best seen by looking down the tube at a white surface.

The liquid at the same time becomes blue from the production in it of copper nitrate :—

$$8HNO_3 + 3Cu = \overline{2NO} + 3Cu(NO_3)_2 + 4H_2O.$$

The NO which is evolved is a colourless gas, but it yields reddish-brown NO_2 by oxidation when it mixes with the air in the upper part of the vessel (44).

539. **Indigo Solution,** if it is poured in a cold and dilute condition into cold nitrate solution, until the mixture becomes faintly but decidedly blue, retains its blue colour unchanged when SO_2 is passed into the liquid (see Note, 545), or when *freshly made* H_2SO_3 solution is added. This reaction distinguishes a nitrate from a chlorate. Compare with paragraph 545

540. **Ignition** converts alkaline nitrate into nitrite . hence if any solid nitrate is heated to redness with fusion mixture ($Na_2CO_3 + K_2CO_3$) on platinum foil for several minutes, a soluble alkaline nitrite is formed. If the foil is allowed to cool and is then boiled in water, the alkaline nitrite will be dissolved.

A portion of the solution may be proved to contain nitrite by the starch test (533).

Another portion may be acidified with HNO_3, and will be found to give no precipitate with $AgNO_3$. Compare with paragraph 547.

541. **The action of Hydrogen** also reduces nitric acid to nitrous acid.

Add dilute H_2SO_4 to some nitrate solution, and then add some freshly-made starch-solution containing KI: no colour will be produced : but when a fragment of Zn is dropped in, and hydrogen is evolved, the liquid will assume a deep blue colour (533).

542. **Fusion of a Nitrate on Charcoal** in the blowpipe-flame causes *deflagration*, that is to say, the surface of the charcoal burns rapidly with a slight hissing noise.

543. **Ignition of a Nitrate of a Heavy Metal,** in a glass tube closed at one end, causes the evolution of a reddish-brown gas with a characteristic smell (NO_2) and of oxygen: $PbN_2O_6 = 2NO_2 + O + PbO$. The NO_2 is recognised by its colour and by its smell, and the O is found by inflaming a glowing splinter of wood. Lead nitrate may be used in trying this experiment.

544. **Hydrogen nitrate, or Nitric acid** (HNO_3), has a strongly acid reaction. It gives with $FeSO_4$ solution a brown ring (537), and with Cu a reddish-brown gas and a blue solution (538). When nitric acid is evaporated with quill-clippings it stains them bright yellow.

CHLORATE (ClO_3).—Use $KClO_3$.

545. **Indigo solution,** if it is added in a cold dilute condition to a *cold* solution of chlorate until the liquid is coloured faintly but distinctly blue, loses its colour as soon as SO_2 is passed into the liquid, or when some *freshly-made* H_2SO_3 solution is added and the mixture is shaken. Compare with paragraph 539.

Note.—Since the presence of H_2SO_4 must be avoided in this reaction, it is well to pass SO_2 gas into the liquid which is being tested. The gas may be most readily obtained from a bottle of liquid SO_2.

546. **Strong H_2SO_4,** when it is poured upon a *little* solid chlorate, becomes orange-red in colour, and when the liquid is shaken it evolves a *bright yellow gas* (Cl_2O_4):—

$$3KClO_3 + H_2SO_4 = \overline{Cl_2O_4} + KClO_4 + K_2SO_4 + H_2O.$$

This gas has a smell somewhat resembling that of chlorine. It undergoes explosive decomposition readily when it is gently heated, and the acid mixture in the tube accordingly crackles and even detonates when it is warmed by the flame.

Caution.—This experiment may be dangerous, unless very little chlorate is used, and the mixture is then heated gently with the mouth of the test-tube turned away from the person.

547. **Ignition of Alkaline Chlorate evolves Oxygen and produces Chloride.**

$AgNO_3$ solution produces no precipitate with solution of chlorate if chloride is absent.

But if some solid potassium chlorate is heated in a test-tube, it will fuse and give off oxygen with effervescence: the chlorate is meanwhile converted into chloride :—

$$KClO_3 = KCl + O_3.$$

The oxygen gas may be detected by holding in the tube a slip of wood with a spark at its end; the spark will burst into a flame.

If the tube is then allowed to cool, and the residue is dissolved by heating it with water, a portion of the solution will yield no evidence of the presence of a nitrite (533).

Another portion of the solution will yield a white precipitate (AgCl) on the addition of $AgNO_3$; this precipitate will not dissolve in HNO_3 even on boiling, but will dissolve readily in AmHO (552): it is therefore produced by the chloride which has been formed from the chlorate, and indirectly proves the presence of chlorate.

A chlorate which does not yield a soluble chloride must be mixed with fusion-mixture before it is heated for this test.

This reaction readily distinguishes a chlorate from a nitrate. Compare with paragraph 540.

548. **When a Chlorate is heated on Charcoal** in the blowpipe-flame, it causes the charcoal to deflagrate.

549. **Hydrogen chlorate, or Chloric acid** ($HClO_3$), is an odourless liquid, which first reddens and then bleaches litmus-paper. When

it is kept for some time, it changes into O, Cl, $HClO_4$ and H_2O. The same change occurs rapidly when the acid is heated.

DETECTION OF NITRATE AND CHLORATE.

550. There is little difficulty in distinguishing nitrate from chlorate.

Nitrate and chlorate, when they are present together, more or less interfere with one another's reactions. But they may be detected in the absence, or after the removal of chloride and nitrite, by heating the solid substance strongly for a short time, and then testing for nitrite and chloride (540, 547).

The preliminary removal of nitrite may be effected by boiling the substance with excess of acetic acid; and chloride may be removed by precipitation with $AgNO_3$, which must, however, not be added in excess. It is evident that the portion of the solution, to which $AgNO_3$ has been added, cannot be used for the test for nitrate.

GROUP IV.—CHLORIDE GROUP.

551. This Group includes chloride, bromide and iodide.

These Acid-radicles closely resemble one another in many of their reactions. They are precipitated by $AgNO_3$ from their solutions, after HNO_3 has been added in excess. In this respect they differ from all acid-radicles except cyanide, ferrocyanide and ferricyanide; and these are easily distinguished by other means.

Chloride (Cl).—Use NaCl.

552. $AgNO_3$, when it is added to the solution of a chloride, gives a *pure white* precipitate (AgCl).

This precipitate collects into curdy masses when the liquid is heated or shaken: it rapidly turns violet in sunlight, and slowly darkens even in diffused daylight.

Decant the water, and warm one portion of the precipitate with HNO_3: it will not dissolve. To another portion of the precipitate add AmHO: it will readily dissolve. AgCl is also soluble in KCy-solution and in $Na_2S_2O_3$-solution.

Decant the liquid and warm the precipitate with a little strong H_2SO_4: the acid will remain colourless, and no violet vapour will be given off as is the case with AgI (**562**).

553. Strong H_2SO_4, when it is added to a solid chloride, causes colourless HCl to be given off: this gas may be recognised by fuming in the air, by reddening moistened blue litmus-paper, and by making a drop of $AgNO_3$-solution, acidified with HNO_3, milky (**505**, 1–4).

554. MnO_2 and H_2SO_4.—Mix together intimately some finely-powdered chloride and MnO_2, then add strong H_2SO_4 and warm the mixture : chlorine gas will be evolved :—

$$2NaCl + MnO_2 + 3H_2SO_4 = 2Cl + MnSO_4 + 2NaHSO_4 + 2H_2O.$$

This gas is recognised by its power of bleaching a piece of moistened litmus-paper, which is introduced into the tube on a glass rod.

The most delicate way of making the test is to mix the substances in a small beaker, and then cover this with a watch-glass which bears on its under surface a piece of moistened litmus-paper ; the mixture is then warmed and allowed to stand for some time, and the test-paper is occasionally examined in order to see if its colour has disappeared.

Moist starch is not coloured when it is held in the escaping gas : this distinguishes chloride from bromide, when they are examined by this test. Compare with paragraph 558.

Many samples of commercial MnO_2 evolve Cl when they are heated with H_2SO_4. Hence, unless the MnO_2 has been specially prepared by precipitation, or is known to give no Cl when it is heated with H_2SO_4, it must be heated with H_2SO_4 as long as any bleaching action is produced on moist litmus-paper held in the tube ; the substance to be tested may then be added, and Cl may be again tested for while the mixture is being heated

555. Formation of CrO_2Cl_2 by the action of H_2SO_4 and Chromate.—An intimate mixture is made of a solid chloride with three or four times as much $K_2Cr_2O_7$, by rubbing the two substances together to a fine powder in a mortar. This mixture is then heated with strong H_2SO_4 in the tube or flask represented in figure 73 (p. 202), or in a Clarke's retort (fig. 74, p. 216) Reddish-brown vapour (CrO_2Cl_2) will be evolved :—

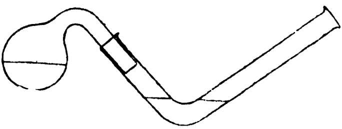
$$4NaCl + K_2Cr_2O_7 + 6H_2SO_4 = 2CrO_2Cl_2 + 4NaHSO_4 + 2KHSO_4 + 3H_2O.$$

If the vapour escaping from the delivery-tube is passed into some water, contained in a test-tube or in the bend of the condenser of Clarke's apparatus (fig. 74), the water will become reddish-yellow in colour, owing to the formation of H_2CrO_4 :—

$$CrO_2Cl_2 + 2H_2O = H_2CrO_4 + 2HCl.$$

When excess of AmHO is added to the reddish-yellow liquid the colour will change to pale yellow, since Am_2CrO_4 is formed : but the original reddish-yellow colour will be reproduced, when excess of $H\bar{A}$ is added; and the presence in this liquid of H_2CrO_4, and therefore of chloride in the original substance, may be proved by the formation of a yellow precipitate ($PbCrO_4$) on the addition of $Pb\bar{A}_2$-solution.

FIG. 74.



CLARKE'S RETORT.

Caution.—The detection of a chloride by this method depends on the formation of H_2CrO_4 by the action of the water in the tube on the vapour of CrO_2Cl_2. Great care must therefore be taken that none of the mixture containing chromate or chromic acid is allowed to pass into the delivery-tube and thence into the water; since if this occurred, chromic acid would be found even when no chloride was present.

Care must also be taken that the water is not sucked back into the hot strong H_2SO_4 when the apparatus shown in fig. 73 is used. This is prevented by raising the delivery-tube out of the water as soon as the heating is stopped.

556. **Hydrogen chloride, or Hydrochloric acid (HCl)**, is a colourless gas, which fumes in the air, and dissolves very readily in water. Both the gas and its solution render $AgNO_3$-solution, which has been acidified with HNO_3, milky. The solution of the acid evolves chlorine gas when it is heated with MnO_2.

BROMIDE (Br).—Use NaBr.

557. **$AgNO_3$**, when it is added to solution of a bromide, gives a *yellowish-white* precipitate (AgBr).

This precipitate is easily coagulated when the liquid containing it is heated or shaken; it is insoluble in HNO_3, easily soluble in KCy-solution and in $Na_2S_2O_3$-solution, and slightly soluble in AmHO. It resembles the precipitate of AgCl in these respects; but it is distinguished from AgCl by its yellowish colour, and by its slight solubility in AmOH.

If the liquid is decanted and the precipitate is heated with strong H_2SO_4, no violet vapour will be evolved as is the case with AgI (562).

558. **Strong H_2SO_4,** if it is heated with a bromide, causes the evolution of pungent acid fumes (HBr), which are accompanied and coloured by *reddish-brown* vapour of Br. This vapour somewhat resembles Cl in smell and by bleaching litmus, but it differs from Cl in colour.

The bromine vapour may also be recognised by its power of staining cold moist starch *orange-red.* The starch-powder may be taken up on the wetted end of a glass rod, and then moistened by breathing upon it several times.

If MnO_2 is mixed with the bromide before H_2SO_4 is added, Br is evolved in greater quantity (554).

559. **Chlorine-water,** or solution of bleaching-powder which has been acidified with HCl, if it is added carefully drop by drop to solution of a bromide, liberates Br, and this colours the solution orange-red.

The addition of Cl in excess must be carefully avoided, since Cl combines with Br and destroys its colour.

Warm a part of the coloured solution; reddish-brown Br vapour will be given off, and will stain cold moist starch *orange-red* (558).

Add carbon bisulphide (CS_2) to another portion of the coloured solution, and mix the liquids well by shaking them; the Br will be dissolved away from the water by the CS_2. If the liquid is allowed to stand at rest for a short time, the reddish-brown solution will sink beneath the colourless water.

If a little KHO solution is now added and the liquids are shaken up together, the colour of the Br will disappear from the CS_2; this is due to the formation of the colourless salts KBr and $KBrO_3$, which remain in solution in the water:—

$$6Br + 6KHO = 5KBr + KBrO_3 + 3H_2O.$$

560. Evolution of Bromine on addition of H_2SO_4 and Chromate.—If an intimate mixture of a solid bromide and $K_2Cr_2O_7$ is heated with strong H_2SO_4 in the apparatus described in paragraph 555, reddish-brown bromine vapour will be evolved.—

$$6NaBr + K_2Cr_2O_7 + 11H_2SO_4 = 3Br_2 + Cr_2(SO_4)_3 + 6NaHSO_4 + 2KHSO_4 + 7H_2O.$$

The Br vapour may be passed into water, and it will colour the liquid reddish-brown.

If CS_2 is then shaken up with part of this brown solution, it will dissolve away the Br and will settle beneath the colourless water as a brown layer.

If another part of the brown aqueous solution of Br is shaken with AmHO or KHO, the colour of the solution will be destroyed.

These reactions serve to detect a chloride in the presence of a bromide. Compare with paragraph 555.

561. **Hydrogen bromide, or Hydrobromic acid (HBr),** is a colourless, soluble gas, which fumes in the air, and closely resembles HCl. Its solution differs from that of HCl by evolving coloured Br vapour when it is heated with MnO_2.

Iodide (I).—Use KI.

562. $AgNO_3$, when it is added to solution of an iodide, gives a *yellow* precipitate (AgI).

This precipitate is easily coagulated when the liquid containing it is heated or shaken: it is insoluble in HNO_3, very slightly soluble in AmHO, but easily soluble in KCy-solution and in $Na_2S_2O_3$-solution.

The precipitate of AgI differs from the precipitates of AgCl and AgBr by its colour and by its very slight solubility in AmHO; but it is distinguished with greater certainty by its behaviour when it is heated with strong H_2SO_4.

If the liquid is decanted from the precipitate, and the precipitate is then heated with strong H_2SO_4, the acid will be coloured red, and violet vapour of iodine will be seen on looking down the tube. The coloured vapour is most distinctly seen after the tube has been allowed to cool for a short time.

If a mere trace of iodine vapour is evolved, it may be detected by holding in the tube a glass rod which has been dipped into freshly-made starch solution; the starch will be coloured blue.

The starch solution is made by stirring a *little* starch powder with some water in a small porcelain dish, then boiling for a few seconds, and cooling the solution.

563. **$CuSO_4$-solution** produces a white precipitate (Cu_2I_2) in solution of iodide, and causes the separation of iodine, which colours the liquid brown :—

$$4KI + 2CuSO_4 = Cu_2I_2 + 2K_2SO_4 + I_2.$$

If the copper sulphate solution has been mixed with solution of H_2SO_3 or of $FeSO_4$ before it is added to the iodide, the precipitate and liquid are not discoloured by free iodine :—

$$2CuSO_4 + 4KI + H_2SO_3 + H_2O = Cu_2I_2 + 2K_2SO_4 + H_2SO_4 + 2HI.$$

The formation of the precipitate is accelerated by warming the liquid.

Separate this precipitate from the liquid by decantation or by filtration, and warm it with strong H_2SO_4; the acid will become red, and violet iodine vapour will be evolved.

Solution of chloride and of bromide differs from that of iodide by not being precipitated by $CuSO_4$-solution.

564. **Strong H_2SO_4,** if it is warmed with a solid iodide, causes the evolution of acid fumes (HI), accompanied by

violet iodine vapour. If the iodine vapour is large in quantity, it will condense on the inside of the tube as a black solid.

The iodine vapour colours starch solution blue. This may be proved by holding in the vapour a glass rod, or a strip of paper, which has been moistened with freshly-made starch-solution; or by pouring out the heavy iodine vapour into a white porcelain dish, the inside of which has been wetted with the starch-solution.

Iodine is evolved in larger quantity, if MnO_2 has been mixed with the iodide before it is warmed with H_2SO_4 (554).

565. **Chlorine-water,** or solution of bleaching-powder which has been acidified with HCl, if it is added carefully drop by drop to solution of an iodide, sets iodine free. The iodine dissolves in the water, colouring it brown: some of the iodine may also separate as a black powder.

The addition of Cl in excess must be carefully avoided, as it would form colourless iodine chloride, which does not give the following reactions.

Divide the brown liquid into three parts in three test-tubes.

1. **Heat** one part; violet iodine vapour will be given off, and will be seen on looking down the tube at a white surface. The colour is most distinctly seen if some strong H_2SO_4 is added to the liquid before it is heated. If a glass rod is dipped into starch-solution and is then held in the iodine vapour, the starch will become blue.

2. To another portion of the brown solution add **starch-solution**; an intense blue colour will be produced. The colour often appears black, unless much starch-solution is added and the liquid is largely diluted. When the solution is heated it becomes colourless, but it often regains its colour on being cooled: the solution must be diluted, and the starch must be added in excess, in order to ensure the removal of the colour by heat.

3. To the third portion of the brown solution add a drop

of CS_2 and shake well; the CS_2 will dissolve the iodine, and this solution will gradually settle down as a beautiful violet layer beneath the water, when the liquids are allowed to stand at rest.

Pour in a little KHO solution, and shake the liquids together; the violet colour of the CS_2 will disappear, owing to the formation of the colourless salts KI and KIO_3.

566. Nitrous acid (HNO_2).—If a little KNO_2 is dissolved in solution of an iodide and a dilute acid is added, HNO_2 is produced and liberates the iodine.

A solution of N_2O_4 in dilute H_2SO_4 also sets free iodine from an iodide (**1152**).

The iodine thus liberated may be identified by the three methods which are described in paragraph **565**; of these the starch test and the CS_2 test are the most delicate.

When the two reagents, mentioned above, are used for liberating iodine from an iodide, they present the advantage over Cl that they do not hinder the detection of the iodine, even when they have been added in excess. They also present the advantage that they do not liberate Br from a bromide.

567. **$HgCl_2$ solution** gives with solution of an iodide a scarlet precipitate (HgI_2), which is easily soluble in excess of solution of $HgCl_2$ or of soluble iodide. With a solution of bromide or chloride, $HgCl_2$ gives no precipitate.

568. **$Pb\bar{A}_2$-solution** gives with solution of iodide a *bright yellow* precipitate (PbI_2) if this precipitate is dissolved in the least possible quantity of boiling water, it separates again in the form of beautiful golden yellow crystals when the liquid is cooled.

With solution of bromide or chloride, $Pb\bar{A}_2$-solution gives a *white* precipitate.

569 **Hydrogen iodide, or Hydriodic acid (HI)**, is a colourless, fuming, soluble gas, resembling HCl and HBr. Its solution differs from solution of HCl or of HBr by evolving iodine when it is heated with MnO_2.

DETECTION OF CHLORIDE, BROMIDE, OR IODIDE, WHEN IT OCCURS ALONE.

570. **Chloride, Bromide, or Iodide** is easily detected by the foregoing tests. The reactions given in paragraphs 554, 555 are perhaps the most characteristic tests for a chloride; and the tests described in paragraphs 558, 559, 560 distinguish a bromide; an iodide is detected with the greatest ease and certainty by the reaction described in 566.

The colour of the precipitate which has been produced by adding $AgNO_3$ to the solution made acid with HNO_3, and the behaviour of this precipitate with AmHO, are less trustworthy indications than the above tests; but they often serve to demonstrate which of these acid-radicles is present. AgCl is *pure white* and is very easily soluble in AmHO; AgBr is *pale yellow* and is not readily soluble in AmHO; while AgI is *primrose-yellow* and is almost insoluble in AmHO.

The action of hot strong H_2SO_4 on the precipitate of AgI, which is described in paragraph 562, is also distinctive of iodide.

DETECTION OF CHLORIDE, BROMIDE, AND IODIDE, WHEN THEY OCCUR TOGETHER.

The detection of these acid-radicles, when they occur together in solution, may be effected by either of the methods which are described in paragraphs 572, 573 The method in par. 572 will be found to be the more rapid in practice.

If chloride is known to be absent, bromide and iodide may be tested for by the method described in paragraph 574.

Insoluble salts must be treated as is directed in paragraph 571,* before they can be examined by the following methods.

571.* **Insoluble Compounds** must be decomposed by means of Na_2CO_3, in order to obtain the chloride, bromide, and iodide in solution.

Boiling with Na_2CO_3-solution will effect this change in the case of some substances, but heating the finely-powdered substance with three times its weight of fusion mixture ($Na_2CO_3 + K_2CO_3$) on platinum foil or in a platinum capsule is a more certain and general method.

The carbonates are fused for about ten minutes; the mass is allowed to cool, and is then boiled with water and the filtered solution is examined by one of the following methods (572, 573, 574).

This treatment with fused alkaline carbonates may be employed for rendering the precipitate which has been produced by $AgNO_3$ (552, 557, 562) suitable for the following examination.

572.* **First Method.**—The most convenient method of examining for the presence of chloride, bromide, and iodide, when they may be present together in an aqueous solution, consists in testing for bromide and iodide in one part of the liquid by the chlorine test (559, 565); then driving off bromine and iodine, if they are present, from another part of the liquid by means of acidified $K_2Mn_2O_8$ solution, and testing for chloride by means of $AgNO_3$ (552).

The aqueous solution of the substance is referred to below as the "original solution." If the substance to be tested is insoluble in water, it must be treated with Na_2CO_3 as is directed in paragraph 571* in order to obtain the acid-radicles in solution.

To a portion of the original solution, which is to be tested, add a few drops of CS_2: then add to it cautiously, drop by drop, either chlorine-water or acidified solution of bleaching-powder, and shake the liquid after each addition.

If the CS_2 does not become Coloured, bromide and iodide are absent; chloride may then be tested for, by adding to another part of the original solution HNO_3 in excess and $AgNO_3$ (552).

If the CS_2 becomes Coloured, note the colour which first appears. If the CS_2 becomes *brown* at once, bromide is present and iodide is absent; if the CS_2 is coloured *violet*, iodide is present, and the cautious addition of the chlorine solution must be continued with shaking; if the CS_2 turns *brown*, the presence of bromide is proved: if no brown colour appears, bromide is absent.

Proceed to test for chloride as is directed below.

Remove bromine and iodine from another portion of the original solution by acidifying with dilute H_2SO_4, adding a little potassium permanganate solution, and boiling the solution in a porcelain dish until the brown colour of the liquid disappears. Then add more permanganate solution cautiously and boil the liquid, and continue these processes until the addition of permanganate no longer produces any brown colour, and the liquid retains a pale pink colour for a few seconds: this proves that all bromide and iodide have been removed.

Now add $AgNO_3$: the formation of a white precipitate will prove the presence of chloride.

573.* **Second Method.**—The method of examination is varied according as iodide is present or absent Since chloride cannot be detected in the presence of iodide, the iodide must be separated before proceeding to examine for the chloride.

To a small portion of the Original Solution, which has been made just acid by the addition of dilute H_2SO_2, add a little cold freshly-prepared starch-solution. Then add, drop by drop, either strong HNO_3, or solution of nitrous acid (566). The appearance of a dark blue coloration will show the *Presence of Iodide.*

Examine the remainder of the original solution for chloride and bromide, according to the directions given under (*a*) or (*b*), the selection of the method depending upon whether iodide has been found or not.

(*a*) *Iodide is Present.*—Add to the original solution a mixture of $CuSO_4$-solution with half its measure of strong H_2SO_3-solution, and warm gently for a short time; Cu_2I_2 will be precipitated (563).

In order to ascertain whether the iodide has been entirely precipitated, filter a small quantity of the liquid and warm it with a little more of the above reagent. If any further precipitate is produced, return the filtered portion to the unfiltered liquid. Then add more of the reagent to the whole of the liquid, warm it again for a time and test it again, in order to see if the removal of iodide is complete. Repeat these trials until a small portion of the filtrate gives no further precipitate, when it is warmed with more of the reagent.

Then filter the liquid until it is perfectly clear, add KHO in excess to the filtrate, and boil the liquid. Filter off the precipitate thus produced; and test the filtrate, which is now free from iodide, for bromide and chloride as is directed below (*b*).

(*b*) *Iodide is Absent.*—Make the solution alkaline, if it is not already so, by the addition of pure KHO; and evaporate it to dryness in a porcelain dish. Scrape the residue off the sides of the dish, and mix it with three or four times as much powdered $K_2Cr_2O_7$ by rubbing the two substances together in the dish with a pestle.

Transfer this mixture to the tube or flask shown in figure 73 (505), or to a Clarke's retort (fig. 74, 555), taking the greatest care to let none of the powder remain in the neck or in the delivery-tube.

Then pour in sufficient strong H_2SO_4 to cover the powder to the depth of about a quarter of an inch, and mix the acid and powder by cautious shaking. Dip the end of the delivery-tube into water contained in a test-tube or receiver and warm the mixture gently, carefully guarding against any of the mixture passing over into the water in the test-tube or receiver.

As soon as no more reddish-brown vapour is visible in the delivery-tube, discontinue the heating, and immediately withdraw the tube containing the water.

If no coloured vapour has been evolved and the water in the tube is colourless, chloride and bromide are absent.

If the water is coloured, pour into it sufficient CS_2 to form a layer about half an inch in depth at the bottom of the test-tube. Close the mouth of the tube with the thumb, shake the tube vigorously, and then allow it to stand at rest. If the CS_2, as it settles down, shows a brown colour, *the Presence of Br, derived from Bromide*, is shown.

Separate the water from the CS_2 by pouring it through a wet filter; add to the filtrate AmHO in excess, then $H\bar{A}$ in excess, and then $Pb\bar{A}_2$-solution. The formation of a yellow precipitate indicates the presence of a chromate, and this proves indirectly that *Chloride was present* (555) in the original substance.

574.* Iodide and Bromide may be tested for in the solution as follows. Pour sufficient CS_2 into the liquid to form a large drop at the bottom. Then add, drop by drop, either dilute chlorine-water or acidified solution of bleaching-powder, shaking thoroughly after each addition.

Iodine will be liberated and will colour the CS_2 purple if *Iodide is Present.*

Continue the addition of Cl-solution very cautiously: the violet colour will disappear, and the appearance of a *brown* coloration in the CS_2 will indicate the *Presence of Bromide.* The brown colour may be destroyed by the further addition of chlorine-solution.

GROUP V.—PHOSPHATE GROUP.

575. **This Group includes** phosphate and arsenate

Phosphate and Arsenate resemble one another closely in many of their reactions. They present points of difference, however, which render their distinction possible.

PHOSPHATE (PO_4).—Use $Na_2HPO_4.12H_2O$.

576. **$MgSO_4$-solution,** to which some AmCl has been added and then a little AmHO, gives with solution of a phosphate a white crystalline precipitate ($MgAmPO_4.6H_2O$.)

This precipitate is almost insoluble in AmHO, but is easily soluble in acids. If very little phosphate is present, the precipitate often appears only when the liquid has been gently warmed, then well stirred or shaken, and then allowed to stand for some time.

577. **Fe_2Cl_6-solution,** if it is dropped into phosphate solution, to which a little $H\bar{A}$ and $Na\bar{A}$-solution have been previously added, gives a yellowish-white precipitate ($FePO_4$).

578. **Ammonium molybdate ($AmHMoO_4$).**—When a drop of phosphate-solution is *added to* a solution of $AmHMoO_4$ in HNO_3, it gives a yellow precipitate.

The formation of this precipitate is hastened by *gently* warming, and stirring or shaking the liquid; but the precipitate appears only after a time in very dilute solution of a phosphate. See *Precautions* on p. 227.

A part of the precipitate usually adheres firmly to the inside of the tube; this may be removed by solution of KHO, NaHO, or AmHO, in which it is readily soluble. The precipitate is only slightly soluble in inorganic acids, and is practically insoluble in HNO_3.

Precautions.—This is a most delicate test if it is properly performed. It serves to detect the presence of mere traces of phosphate, when careful attention is paid to the following precautions.

The $AmHMoO_4$-solution must be prepared according to the directions given in paragraph 1162, Remark 40.

The solution to be tested must not be alkaline to test-paper It should be made distinctly acid by addition of HNO_3, and then added *in small quantity only* to some of the $AmHMoO_4$-solution in a test-tube. More of the solution must only be added, if no yellow precipitate forms after the liquid has been *gently* warmed and stirred.

This last precaution is extremely important, since the presence of *an excess of phosphate altogether prevents the formation of the precipitate.*

Show that this is the case, by pouring a few drops of $AmHMoO_4$-solution into some Na_2HPO_4-solution which has been acidified with HNO_3. No precipitate will form even when the liquid is heated and shaken, since the phosphate is present in large quantity as compared with the $AmHMoO_4$. If a few drops of this liquid mixture, however, are now added to some fresh $AmHMoO_4$-solution, the precipitate will appear.

It must also be remembered that the presence of much HCl retards or prevents the formation of this precipitate. A solution of the substance acidified with nitric acid alone, should therefore be used if possible.

579. **$AgNO_3$** : yellow precipitate (Ag_3PO_4) : pour off a portion of the liquid with the precipitate and show that the precipitate is soluble in AmHO and in HNO_3.

580. **Ignition with Mg or Na** —A very delicate test for the presence of P, occurring in the form of phosphate or otherwise, consists in strongly heating the dry solid substance with Mg powder, or with a small piece of Na, in a little tube of hard glass closed at one end. When the cool tube is broken and the powdered substance is breathed upon, a smell of onions will be perceived ; this is due to the production and escape of PH_3, which has been formed by the action of the moisture of the breath upon the metallic phosphide.

581. **Flame-Coloration.**—If a phosphate is made into a paste with strong H_2SO_4 and the mixture is then heated strongly at the tip of the inner blowpipe-flame, it gives a *bluish-green* colour to the flame.

582. **Blowpipe-test.**—Many phosphates, when they are moistened with $Co(NO_3)_2$-solution, and are then heated in the outer blowpipe-flame, become *blue*.

583. **Hydrogen phosphate, or Phosphoric acid (H_3PO_4),** is a colourless crystalline substance Its solution is strongly acid, and differs from H_2SO_4 by not charring paper which has been dipped into it and then dried by heat. The presence of H_3PO_4 may be detected by reactions 576, 578.

ARSENATE (AsO_4).—Use Na_2HAsO_4 $12H_2O$ solution.

584. The precipitates formed in reactions 576–578 by a phosphate, are precisely similar in appearance and general properties to those formed when the same reagents are used with solution of an arsenate. But $AmHMoO_4$ gives a precipitate with an arsenate *only when the liquid is boiled*, and not when it is *gently heated*, as in the case of a phosphate.

Moreover, the precipitate which $AgNO_3$ gives with an arsenate is *brown*, while that given by a phosphate is *yellow* (579).

DETECTION AND SEPARATION OF ARSENATE AND PHOSPHATE.

585. **The following Differences** serve to detect and separate phosphate and arsenate:—

1. The fact that an arsenate yields a precipitate with $AmHMoO_4$ only when the liquid is *boiled*, whereas the corresponding precipitate is produced by a phosphate when the liquid is only *gently heated.*

2. Arsenate solution, which has been boiled with strong HCl, gives, when H_2S is passed into the hot liquid, first a white precipitate of S and then yellow As_2S_3; phosphate solution under the same circumstances gives no precipitate.

3. In *perfectly neutral* arsenate solution $AgNO_3$ gives a *brown* precipitate; with phosphate it gives a *canary-yellow* precipitate.

DETECTION OF PHOSPHATE OR ARSENATE.

586. The Simplest Method of ascertaining which of these two Acid-radicles is present is to throw the precipitate, which has been produced by the addition of AmCl, AmHO and $MgSO_4$ (576) upon a filter; then wash it with a little cold water, and drop $AgNO_3$-solution upon it. If the precipitate consists of $MgAmAsO_4$, it will become *brown;* if it is $MgAmPO_4$, it will become *canary-yellow.*

The precipitate, which has been produced by the addition of AmCl, AmHO and $MgSO_4$, may also be examined for arsenate by dissolving a part of it in dilute HNO_3, adding $AgNO_3$-solution as long as it causes any white precipitate, and then adding *very dilute* AmHO gradually drop by drop.

If arsenate is present, a *brown* precipitate will form just before the liquid becomes alkaline to test-paper: a phosphate will give a *yellow* precipitate.

Instead of mixing the dilute AmHO with the acid liquid, the AmHO may be gently poured in upon the top of the liquid: the colour will then appear at the surface of contact of the ammonia-solution with the acid liquid.

DETECTION OF ARSENATE, PHOSPHATE AND ARSENITE, WHEN THEY ARE PRESENT TOGETHER.

587.* A clear mixture of solutions of $MgSO_4$, AmCl and AmHO is added as long as it causes any further precipitate: the liquid is filtered and the filtrate is preserved.

A small portion of the precipitate is then tested at once for arsenate by dropping $AgNO_3$-solution upon it. If the precipitate turns yellow, *Phosphate is present, and Arsenate is absent.* If the precipitate turns brown, *Arsenate is present,* and Phosphate may also be present.

The arsenate must be separated from the other part of the precipitate before phosphate can be tested for.

The rest of the precipitate is accordingly dissolved in a little boiling strong HCl, and H_2S is passed for several minutes into this hot solution ; the presence of an arsenate will be confirmed by the precipitation of white S and yellow As_2S_3 (313).

This precipitate is filtered off and H_2S is again passed into the boiling filtrate. If any further precipitate is produced, the gas must be further passed until it no longer causes a precipitate in the boiling liquid.

The precipitation by H_2S is rendered complete without delay, if the original solution in HCl is first treated with H_2SO_3. See paragraph 313.

The clear filtrate is then evaporated just to dryness, the residue is dissolved in a little dilute HNO_3, and this solution is tested for phosphate by adding a few drops of it to $AmHMoO_4$ solution and gently warming the liquid (578).

Arsenite may be tested for by acidifying the filtrate, which has been reserved from the original Mg precipitate, and passing H_2S into it. Yellow As_2S_3 will be precipitated at once if arsenite is present.

VI.—UNGROUPED ACID-RADICLES.

The following Acid-radicles are not included in any of the preceding Groups :—Borate, Chromate, Silicate, Fluoride, Fluosilicate, Permanganate, Phosphite, and Hypophosphite; together with the rarer Acid-radicles, Tungstate, Molybdate, Selenite, Tellurate, Tellurite, Titanate and Vanadate.

These Acid-radicles present no marked resemblances to one another or to other Acid-radicles in their reactions; they are therefore usually detected by special tests. The reactions of the rarer acid-radicles have been already described under the metals, since they are precipitated by Group-reagents for the metals.

BORATE (BO_3).—Use Borax, $Na_2B_4O_7 10H_2O$.

588. Turmeric-test.—Powder some borate finely, and stir it with a little dilute HCl on a watch-glass. Immerse the lower half of a strip of turmeric-paper in this liquid, and dry it on a watch-glass in the steam-oven (98), upon a water-bath (88), or above a small flame. The part of the test-paper which was moistened will now appear *reddish-brown*, and will become *bluish-black* when it is moistened with AmHO.

589. Alcohol-flame Coloration.—Pour alcohol upon some finely-powdered borate in a test-tube or porcelain dish: add a little strong H_2SO_4; then heat the mixture, and kindle the spirit. The flame will show a *green edge;* this is often most clearly seen when the vapour is relighted after the flame has burnt for a time and has then been extinguished.

This flame-coloration is similar to that which is given by certain other substances; but its spectrum, consisting of three or four equidistant green lines, is quite characteristic (fig. 68, p. 100).

590. **Blowpipe-flame Coloration.**—If a mixture containing CaF_2, $KHSO_4$, and a borate is finely powdered, then moistened, and heated on a loop of platinum wire in the inner blowpipe-flame for a short time, it will colour the outer flame momentarily *green.*

This test is not always decisive, since many phosphates and copper salts give a similar result. The flame-coloration is also liable to be masked by the colorations of other substances.

591. **Hydrogen borate, or Boric acid (H_3BO_3),** usually occurs combined with $2H_2O$ as a crystalline scaly hydrate. When this substance is strongly heated, it yields a fusible glass consisting of B_2O_3.

The solution of boric acid turns the colour of blue litmus-paper to wine-red.

The reactions in paragraphs 588, 589, and 590 serve to detect boric acid.

The acid is soluble both in water and in alcohol; and when these solutions are evaporated, the boric acid partially escapes with the vapour of the liquid.

CHROMATE (CrO_4). —Use Potassium dichromate, $K_2Cr_2O_7$.

All chromates are more or less yellow or red in colour. The yellow solution of a normal chromate becomes orange-red when an acid is added, owing to the formation of a dichromate; while the reddish solution of a dichromate becomes pale yellow when it is made alkaline, owing to its conversion into a normal chromate. See remarks under Chromium, p. 137.

592. **Reduction of Chromate to Chromic salt.**—Various substances, which readily combine with oxygen, deoxidise acidified chromate solution and produce a chromic salt; the colour of the solution at the same time changes from *orange-red to bright green.*

Add HCl or H_2SO_4 to some chromate solution, and deoxidise separate portions of this liquid by each of the following methods. In every case the colour of the liquid will change to bluish-green.

(a) Pass H_2S into the hot solution; white S will separate owing to oxidation of the hydrogen of the H_2S.

(b) Add $NaHSO_3$ or H_2SO_3 and warm.

(c) Add alcohol and boil : the smell of aldehyde will be noticed.

(d) Boil for some time, after adding much strong HCl : Cl gas will be slowly evolved.

(e) Add Zn and warm : the reducing action of the nascent H will be slow.

The following equations show the reactions which occur with H_2S and with HCl —

$$K_2Cr_2O_7 + 3H_2S + 8HCl = Cr_2Cl_6 + 2KCl + 7H_2O + S_3.$$
$$K_2Cr_2O_7 + 14HCl = Cr_2Cl_6 + 2KCl + 7H_2O + 3Cl_2.$$

The equations representing the other processes of deoxidation may be drawn out by the student.

The green solution, which results from the above reactions, will give all the reactions for a chromic salt (202–205) and since chromic acid is reduced by boiling HCl and by H_2S to chromic salt, the presence of a chromate will lead to the precipitation and detection of $Cr_2H_6O_6$ in the ordinary course of analysis. This chromic hydroxide, however, will be known from the colour of the original solution to be the result of the reduction of chromate.

593. **Hydrogen peroxide** (**H_2O_2**) **or Barium peroxide** (**BaO_2**), if it is added to a *cold* acidified solution of chromate, produces an intense but evanescent *blue coloration.*

This colour is far more permanent, if ether is first added and the liquid is then vigorously shaken immediately after the addition of the peroxide : the ether assumes and retains a blue colour. The addition of a few drops of strong HNO_3 is useful. This reaction is extremely delicate and characteristic.

594. **$Pb\bar{A}_3$** : *yellow* precipitate ($PbCrO_4$) : soluble in KHO, insoluble in $H\bar{A}$.

595. **$AgNO_3$** : *purple-red* precipitate (Ag_2CrO_4) : soluble in AmHO and in HNO_3.

596. **$BaCl_2$** : yellowish-white precipitate ($BaCrO_4$) : insoluble in $H\bar{A}$, soluble in HCl.

597. **Hydrogen chromate, or Chromic acid,** is present in the reddish-yellow liquid which is produced by dissolving CrO_3 in water, and by acidifying a solution of a chromate. Its solution evolves Cl and becomes green, when it is boiled with HCl. When solution of chromic acid is neutralised by an alkali it becomes yellow, and the yellow solution yields the above reactions for chromate.

SILICATE (SiO_3).—Use solution of Sodium silicate, Na_2SiO_3; and finely-ground sand, SiO_2, for a solid.

598. **HCl,** if it is added drop by drop to solution of a silicate, which is meanwhile constantly stirred or shaken, will give a gelatinous precipitate of orthosilicic acid (H_4SiO_4) unless the solution is very dilute. Since the silicic acid may remain in solution, the non-appearance of a precipitate is no proof of the absence of a silicate.

The solution of silicate, which is used for these reactions (No. 146, 1164), is made of such strength that acids do not cause a precipitate in it; but if some of the solution is boiled down to a quarter of its volume, the precipitate will usually be obtained on the addition of an acid.

599. **Separation of SiO_2.**—If silicate is present in small quantity, or is in dilute solution, it may be detected by acidifying the solution with HCl and evaporating the liquid to dryness in a porcelain dish. The evaporation is finished on the water-bath, and the residue is heated on the water-bath (88) as long as acid fumes are given off.

Insoluble SiO_2 is thus produced, and remains undissolved as a white powder when the dry residue is stirred and warmed with strong HCl.

During the last part of the evaporation over a flame, after the silica has separated as a gelatinous mass, spirting will occur unless the heat is lessened and the residue is stirred with a glass rod. This spirting may be also prevented by finishing the evaporation upon a water-bath (88) as soon as the liquid thickens.

The SiO_2, which is left undissolved by HCl, is invisible as long as the liquid remains in the porcelain dish. It is rendered visible by stirring the liquid and at once pouring it out into a glass vessel.

600. **Microcosmic-bead.**—When either a silicate or SiO_2 is fused into a bead of *microcosmic salt* ($NaAmHPO_4.4H_2O$) it is not wholly dissolved, but the SiO_2 floats about in transparent particles in the melted bead, and is visible as little opaque masses when the bead is cold. The particles of SiO_2 are usually most easily seen while they are in motion in the fused bead.

601. **Na_2CO_3 in Blowpipe-flame.**—If solid SiO_2 or a silicate is heated in a fused bead of Na_2CO_3, it causes frothing: this is due to the evolution of CO_2 —$Na_2CO_3 + SiO_2 = CO_2 + Na_2SiO_3$

602. **AmCl, or Am_2CO_3,** causes a gelatinous precipitate of H_4SiO_4, which usually forms only after a time.

603. Certain silicates, if they are moistened with $Co(NO_3)_2$ and are then heated on charcoal in the outer blowpipe-flame, become blue.

604. **Hydrogen silicate, or silicic acid** (H_4SiO_4), can exist in solution in water, and has a slightly acid reaction. It is ordinarily met with, however, in the hydrated state as a colourless gelatinous substance, which does not affect the colour of litmus.

In this gelatinous form silicic acid is insoluble in water, and is only very slightly soluble in acids, but it dissolves as an alkaline silicate in solution of caustic alkali or of alkaline carbonate.

When the gelatinous hydrated acid is strongly heated, it yields a white insoluble powder, SiO_2, which gives the reactions in paragraphs 600, 601.

FLUORIDE (F).—Use finely-powdered CaF_2.

The ordinary tests for a fluoride depend upon the liberation of hydrofluoric acid, which is then allowed to act upon a glass surface and to *etch* or corrode it.

605. **Strong H_2SO_4,** if it is warmed with a little *finely-powdered* fluoride in a test-tube, liberates HF. This acid etches the glass, and causes the sides of the tube to throw off the liquid when it is shaken up, as a greasy surface throws off water.

If the cooled mixture is washed out of the tube and the inside of the tube is perfectly dried, the glass will be seen to be dimmed and roughened. Gentle friction with a glass rod will often detect the roughness of the etched surface, even when this is not perceptible to the eye.

606. Etching a Watch-glass.—The preceding test becomes much more delicate if it is carried out as follows.

A watch-glass is carefully heated by holding it in crucible-tongs at some distance above a flame. Its convex surface is then rubbed with a piece of paraffin-wax, and is thus covered with a melted layer. This layer is made uniform by once more heating the glass, allowing any excess of wax to drop off, and then keeping the glass moving while it cools. The coating of wax is then finally hardened by immersing the glass for a short time in cold water.

Lines or characters are now traced in the wax, near the middle of the glass, by gentle pressure with the point of a penknife; the object being simply to remove the wax from these parts, and not to scratch the glass.

When the glass has been thus prepared, it is placed, with its coated surface downwards, as a cover to a platinum crucible or small leaden cup, which contains a little finely-powdered fluoride and strong H_2SO_4.

Some cold water is then poured into the glass in order to keep it cool, and the bottom of the vessel is heated by a very small flame; the covered vessel is then allowed to stand for about fifteen minutes.

The watch-glass is now removed, and is cleansed by holding it with crucible-tongs above the flame and quickly wiping off the melted wax.

On the clean bright surface thus obtained, the traced lines will usually be visible at once when the glass is held up to the light; but if mere traces of fluoride were present, the tracing may only become visible when the cold surface is breathed upon, and is then examined by reflected light.

607. Formation of Silicon fluoride (SiF_4).—The above methods do not serve for the detection of fluoride in the presence of silicate or of silica, since under these circumstances the action of H_2SO_4 liberates SiF_4 instead of HF.

SiF_4 possesses no power of etching glass. But it gives rise to white fumes in moist air, and when it is passed into dilute AmHO, it yields a colourless flocculent precipitate of H_4SiO_4, and produces AmF in the solution.

The formation of the precipitate of silicic acid is sufficient proof of the presence of a fluoride; but after this precipitate has been filtered off, the AmF may also be detected in the filtrate. This is effected by adding $CaCl_2$-solution to the clear liquid, then filtering off the precipitate of CaF_2, and drying and examining it as is directed in paragraph **606.**

The above method is employed for detecting fluoride in minerals which contain silica or silicate. The method is also sometimes utilised for the detection of fluoride in a substance, which has been previously intimately mixed with SiO_2 so as to render it suitable for the above test.

In order to try the reaction, a mixture of CaF_2 and SiO_2 may be heated with strong H_2SO_4. A small piece of marble should be dropped into the tube in which the SiF_4 is being produced: this evolves CO_2, which carries the silicon fluoride over into the AmHO solution. The changes which occur are thus expressed by equations:—

$$2CaF_2 + SiO_2 + 2H_2SO_4 = SiF_4 + 2H_2O + 2CaSO_4.$$
$$SiF_4 + 4AmHO = H_4SiO_4 + 4AmF.$$

608. $CaCl_2$, when it is added to solution of fluoride, gives an almost transparent gelatinous precipitate (CaF_2), which becomes more visible when the liquid is heated or when AmHO is added: this precipitate is slightly soluble in HCl, but is almost insoluble in $H\bar{A}$. Difference from fluosilicate.

Solution of NaF may be prepared for the above reaction, by fusing a mixture of powdered CaF_2 and fusion-mixture on platinum foil for several minutes · then boiling the cool mass on the foil in water, filtering the solution, and acidifying the filtrate with $H\bar{A}$.

609. Hydrogen fluoride, or Hydrofluoric acid, HF, is at ordinary temperatures a colourless gas, which fumes in the air, etches a glass surface, and dissolves freely in water. The solution of HF is acid in reaction, and differs from all other acids by decomposing and dissolving SiO_2.

FLUOSILICATE (SiF_6).—Use Hydrogen fluosilicate, H_2SiF_6.

610. $BaCl_2$: white precipitate ($BaSiF_6$). This precipitate usually appears only when the solution is gently warmed, and differs in this respect from $BaSO_4$.

It differs from $BaSeO_4$ (370) by being only slightly soluble in boiling HCl: it is insoluble in alcohol.

Allow this precipitate to settle and reserve it for the reaction in paragraph 612.

The above precipitate shows some resemblance to $BaSO_4$ since it is only slightly soluble in HCl, but it differs in appearance by being more or less transparent and crystalline. Fluosilicates differ also from sulphates by giving no precipitate with $Sr(NO_3)_2$ or with $Pb(NO_3)_2$. Further, when they are heated with excess of AmHO they yield a flocculent precipitate of H_4SiO_4, and leave AmF in solution (607).

Fluosilicates also differ from sulphates by the following very distinctive reactions.

611. KCl-solution, when it is warmed with solution of fluosilicate, gives a very transparent gelatinous precipitate (K_2SiF_6): this precipitate is only visible after it has been allowed to settle for a time: it is insoluble in alcohol.

612. Strong H_2SO_4, if it is heated with solution of fluosilicate, causes HF to be given off: this vapour may be detected by its etching action on glass (606).

This reaction is especially useful for ascertaining whether a precipitate, which has been produced by $BaCl_2$, consists of or contains $BaSiF_6$.

The precipitate from paragraph 610 may be tested by this process. The precipitate is allowed to settle: almost all the water is then poured off: the remainder of the water is shaken up with the precipitate and is at once poured into a watch-glass the rest of the water is then decanted, and the precipitate which remains on the glass is warmed for some time with strong H_2SO_4. After the glass has been washed and dried, it will be found to be corroded where the precipitate rested.

When the more delicate process described in paragraph 606 is employed, the precipitate should be shaken up with the liquid and poured off into a platinum crucible or leaden cup. The liquid is then decanted, and the precipitate is warmed with strong H_2SO_4 as is described in paragraph 606.

The processes of filtering off and drying the precipitate may be substituted for decantation, but they are less rapidly executed, and are only suitable when the precipitate of $BaSiF_6$ has been obtained in some quantity.

613. **$CaCl_2$** gives no precipitate in fluosilicate solution. (Difference from fluoride.)

614. **Hydrogen fluosilicate, or Fluosilicic acid, H_2SiF_6**, is a strongly acid liquid, which volatilises entirely as $2HF + SiF_4$ when it is heated in a platinum vessel. Accordingly when this acid is evaporated in a watch-glass, the glass is etched. The acid is precipitated by $BaCl_2$ and by KCl (610, 611).

PHOSPHITE (PHO_3).—Use solution of Na_2PHO_3.

615. **$AgNO_3$**: black precipitate (Ag), when the liquid is allowed to stand or is heated. The addition of AmHO in small quantity increases the delicacy of this test, but it must be remembered that the original precipitate is soluble in excess of this reagent.

616. **$HgCl_2$**: white precipitate (Hg_2Cl_2), which becomes grey (Hg) when the liquid in which it is suspended is heated.

617. **$Pb\bar{A}_2$**: white precipitate ($PbPHO_3$), which is insoluble in acetic acid.

618. **Reduction reactions.**—Solution of phosphorous acid, or of a phosphite which has been acidified with $H\bar{A}$, acts as a reducing agent: but its action is not so powerful as that of hypophosphorous acid (623), as the following reactions will show:—

(a) A drop of **$K_2Mn_2O_8$**-solution is not reduced and decolorised by phosphorous acid; but reduction, attended with loss of colour, takes place when the liquid is heated.

(b) A drop of **$CuCl_2$**- or of **$CuSO_4$**-solution is not reduced and decolorised even when the liquid is heated.

619. **Solid Phosphite,** when it is heated, gives off a mixture of hydrogen and hydrogen phosphide : the latter gas is known by its garlic smell : the mixture of gases is spontaneously inflammable in contact with air, and burns with a bright white flame, emitting white fumes . red phosphorus is usually deposited on the interior of the ignition-tube during this combustion.

HYPOPHOSPHITE (PH_2O_2).—Use solution of $NaPH_2O_2$.

620. **$AgNO_3$** white precipitate ($AgPH_2O_2$), which gradually becomes black ; the change is more rapid when the liquid is heated, and is due to the formation of metallic silver.

621 **$HgCl_2$** white precipitate (Hg_2Cl_2), which forms slowly on standing, but more rapidly when the liquid is heated.

622. **$Pb\bar{A}_2$** : no precipitate. (Difference from phosphite)

623. **Reduction reactions.**—Solution of hypophosphorous acid, or of a hypophosphite which has been acidified with $H\bar{A}$, acts as a powerful reducing reagent . this is proved by the following reactions :—

(a) A drop of **$K_2Mn_2O_8$**-solution is reduced and decolorised, when it is added to the cold solution.

(b) When the solution is mixed with a little **$CuSO_4$**-solution and the mixture is heated to 55° C., red copper hydride (CuH_2) is precipitated, and this evolves H and leaves metallic copper when the liquid is heated.

(c) When the solution is mixed with a little **$CuCl_2$**-solution, the copper salt is decolorised and white Cu_2Cl_2 is precipitated ; the Cu_2Cl_2 then gradually changes to red metallic copper.

624. **Solid Hypophosphite,** when it is heated, gives off hydrogen phosphide ; this gas may be recognised by its garlic smell, and by kindling spontaneously in contact with the air : it burns with a bright white flame which emits white fumes and usually deposits red phosphorus on the interior of the ignition-tube.

PERMANGANATE (″Mn_2O_8).—Use solution of $K_2Mn_2O_8$.

Solid permanganates are usually very dark red or purple in colour ; their solutions are usually purple or reddish-purple.

625. **KHO,** if it is added in large excess, causes the purple colour of

the solution to change to green · this is due to the conversion of the permanganate into manganate ·

$$K_2Mn_2O_8 + 2KHO = 2K_2MnO_4 + H_2O + O.$$

The purple colour reappears when this alkaline green liquid is acidified (629).

626. **Reducing reagents.**—The addition of SO_2, H_2S, acidified solution of nitrite, or of other reducing reagents, at once destroys the purple colour of permanganate-solution, and a nearly colourless manganese salt is formed. It follows that in the ordinary course of analysis permanganate will be converted into a manganous salt, which will be detected in Group III B.

627. **Solid Permanganate**, or permanganate in strong solution, when it is mixed with dilute H_2SO_4, evolves oxygen gas: with dilute HCl chlorine gas is evolved with strong acids, gas is evolved with explosive violence.

MANGANATE ($'MnO_4$) —Use alkaline solution of K_2MnO_4.

Solid manganates are almost black: their solutions are bluish-green in colour.

628. **H_2O.**—Manganates undergo decomposition in contact with water with production of purple permanganate and MnO_2, unless much alkaline hydrate is present —

$$3K_2MnO_4 + 2H_2O = K_2Mn_2O_8 + 4KHO + MnO_2$$

629. **An acid**, if it is added in sufficient quantity, causes the green solution of manganate to change to reddish-purple solution of permanganate . even CO_2 can produce this change, and it may therefore be brought about by exposure of the manganate solution to air:—

$$3K_2MnO_4 + 3H_2SO_4 = K_2Mn_2O_8 + 2K_2SO_4 + MnSO_4 + 3H_2O + O.$$

The above change is reversed when solution of an alkaline hydrate is added in excess to the red solution (625).

630. **Reducing reagents** decolorise an acidified solution of manganate and produce manganous salt (626).

PERSULPHATE ($''S_2O_8$).—Use Ammonium persulphate, $Am_2S_2O_8$, dissolved in *cold* water.

630a. **When a strong cold solution** is boiled it gives off oxygen gas which may be collected and identified by causing a glowing wooden splinter to burst into flame. The persulphate is reduced and sulphate remains in solution.

630b. **$BaCl_2$** gives a precipitate only when the solution is boiled. Since, however, persulphate almost invariably contains sulphate as a product of decomposition, the test should be made by adding $BaCl_2$ in excess, filtering off any $BaSO_4$ through a double filter paper, and then boiling the clear filtrate · the formation of a white precipitate will indicate the presence of persulphate.

630c. **$MnSO_4$-solution** gives a brown precipitate of hydrated MnO_2 when the liquid is boiled.

TUNGSTATE (WO_4).—Paragraphs 397–400.

MOLYBDATE (MoO_4).—Paragraphs 357–363.

SELENITE (SeO_3).—Paragraphs 365–368, 371, 372

TELLURATE (TeO_4).—Paragraphs 377, 378.

TELLURITE (TeO_3).—Paragraphs 374–376, 378.

TITANATE (TiO_3).—Paragraphs 263–267.

VANADATE (VO_4).—Paragraphs 271–276

SECTION IV.—PART IV.

THE DETECTION AND ANALYTICAL REACTIONS OF ORGANIC SUBSTANCES.

Introductory Remarks.—Organic substances may be defined as chemical compounds containing carbon.

Methods are described below for detecting the presence in organic substances of the elements carbon, hydrogen, nitrogen, sulphur, phosphorus, chlorine, bromine, and iodine.

The processes of determining the melting-point of a readily fusible solid and the boiling-point and specific gravity of a liquid, and the separation of liquids of different boiling-points by fractional distillation are also given.

The special analytical reactions for certain important organic substances are then described in detail. These include organic acid-radicles, alkaloids, hydrocarbons, haloid compounds, alcohols and phenols, aldehydes and ketones, amido-compounds, carbohydrates, albuminous substances, and chemically related substances.

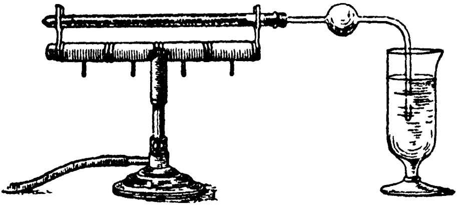
I.—DETECTION OF ELEMENTARY CONSTITUENTS OF ORGANIC SUBSTANCES.

DETECTION OF CARBON.

631. Ignition of Substance.—The presence of carbon in an organic substance is frequently indicated, by the substance becoming charred or blackened, when it is heated alone or with strong H_2SO_4. Carbon is also evolved as CO_2 when certain organic substances are heated either alone or with strong H_2SO_4: the CO_2 thus evolved may be detected by means of lime-water (505).

632. **Ignition with Copper oxide.**—A more certain and general method of detecting carbon consists in mixing the organic substance intimately with finely-powdered copper oxide (CuO), or with some other solid oxidising compound,

FIG. 75.



DETECTION OF CARBON AND HYDROGEN IN AN ORGANIC SUBSTANCE.

and heating the mixture strongly. The carbon is then evolved as CO_2, and this may be detected by passing it into lime-water (505).

The method may be tried by mixing a little sugar with four or five times its measure of finely-powdered CuO. This mixture is then placed in the closed end of a hard glass test-tube, and the remainder of the tube is nearly filled with granular CuO. The open end of the test-tube is then fitted with a bent glass delivery-tube, and the tube is heated in a Bunsen-flame, care being taken that the granular oxide is heated to redness before the mixture itself is heated. The experiment may be made in a longer tube, which is heated by the Ramsay burner (fig. 75): the heat is first applied to the granulated oxide, and when this is at a red heat the mixture is gradually heated to the same temperature. The delivery-tube dips into a vessel containing lime-water, which serves to indicate the evolution of CO_2.

Note 1.—In the case of a volatile liquid the apparatus must be modified so as to cause the vapour of the liquid to pass slowly over the red-hot copper oxide, the products of combustion passing on into the lime-water.

Note 2.—It must be remembered that many carbonates, and all bicarbonates, evolve CO_2 when they are heated. Carbonates may be distinguished from other organic bodies by evolving CO_2 when they are acted upon by cold dilute acid (505).

DETECTION OF HYDROGEN.

633. **Ignition of Substance.**—Many organic substances, which contain oxygen, evolve their hydrogen more or less completely in the form of steam, when they are heated. If the substance is heated in a test-tube, the steam condenses on the cooler part of the tube in the form of water drops.

634. **Ignition with Copper oxide.**—The presence of hydrogen in an organic substance may, however, be detected with greater certainty at the same time as the carbon, by the method of oxidation with CuO at high temperature (632). The hydrogen is converted by this oxidation process into steam, which condenses in the form of water drops in the cooler part of the test-tube, or in the delivery-tube. The bulb in the delivery-tube (fig. 75) is of advantage, since it reduces the velocity of the current of escaping gas and vapour, and therefore promotes the condensation of the steam.

The presence of hydrogen in sugar is proved by the production of the water, which has condensed in the apparatus during the test for carbon (632).

This test is rendered more delicate by placing some white anhydrous $CuSO_4$ in the bulb; the entrance of even a trace of moisture turns the $CuSO_4$ blue.

Note.—Any moisture which is present in the substance or in the copper oxide will be evolved as steam, and will be condensed as is described above. Both the organic substance and the CuO must therefore be perfectly dried, in order to avoid a wrong inference being drawn as to the presence of hydrogen in the organic substance itself. A solid organic substance may usually be freed from moisture by heating it in the steam-oven (98) for about an hour, and the copper oxide should be dried by igniting it to redness for an hour.

DETECTION OF NITROGEN.

635. **Ignition of Substance.**—Nitrogen is evolved from many organic substances in the form of ammonia, or of an organic derivative of ammonia, when the substance is heated. The ammonia, or other alkaline substance, may be readily detected by its pungent smell or by its alkaline action upon moist turmeric-paper (145). See Note (636).

636. **Ignition with Soda-lime**—A more certain and general method of detecting the presence of nitrogen consists in mixing the organic substance intimately with powdered soda-lime, and then heating the mixture strongly in a hard glass test-tube. Either ammonia, or a basic substance containing nitrogen such as an amine, will be evolved, and may be detected by turning moist turmeric-paper brown.

In trying the reaction urea or albumen may be employed.

This test is applicable to most organic substances which contain nitrogen, with the exception of nitro-, nitroso-, azo-, and diazo-bodies.

Note—It must be remembered that many ammonium compounds evolve NH_3 when they are heated, and that they all evolve NH_3 when they are heated with soda-lime. Ammonium compounds, however, evolve NH_3 when they are gently heated with *solution of* KOH; they may be distinguished in this way from most organic compounds containing nitrogen, which require to be heated much more strongly with *solid* KOH, or with soda-lime, before they evolve NH_3.

637. **Ignition with Sodium and Production of Prussian Blue.**—Another general method for the detection of nitrogen consists in converting the nitrogen of the organic substance into a soluble cyanide, and then producing "Prussian blue" from this.

Heat the nitrogenous substance (urea, or albumen) strongly with a small piece of sodium or potassium in a small hard glass ignition-tube. Break up the tube in some water in a mortar with the pestle. Transfer the liquid with the substance to a boiling-tube, warm gently and filter. Then add a few drops of solutions of Fe_2Cl_6 and of $FeSO_4$: and unless the liquid is already strongly alkaline to test-paper, add KOH in excess. Then warm the liquid, and add HCl in excess. The formation of a dark blue precipitate, or of a blue or dark green solution, indicates the formation of Prussian blue; and the nitrogen, which is a constituent of this Prussian blue, must have been derived from the organic substance.

This reaction is more delicate than the preceding one (636); and it serves for the detection of nitrogen in all organic substances with the exception of the diazo-compounds.

Note.—If sulphur is present, the organic substance must be mixed with iron filings, or better with "reduced iron," before it is heated with sodium.

The presence of ready formed cyanogen-compounds in the organic substance may be ascertained by special tests (650 and *Note*).

DETECTION OF SULPHUR.

The Presence of Sulphur in an organic substance may be detected by converting the sulphur either into Na_2S (638) or into Na_2SO_4 (639).

638. Ignition with Sodium, and Formation of Sulphide.—Ignite the organic substance (albumen) with a small piece of sodium: then extract the residue with warm water, filter the solution, and add to the filtrate, made alkaline if necessary with NaOH, solution of sodium nitroprusside: the presence of sulphur will be indicated by *a violet coloration.*

Addition of lead acetate-solution will produce black PbS; but this test is less delicate than the nitroprusside reaction.

639. Ignition with $NaNO_3$, and Formation of Sulphate.—Mix the solid organic substance (albumen) in a porcelain crucible with four times its measure of dry Na_2CO_3 and its own measure of $NaNO_3$, and heat until the alkali salts are fused. Dissolve the cool product in dilute HCl, and add $BaCl_2$: *a white precipitate* ($BaSO_4$) shows the presence of sulphate, which has been formed from sulphur in the organic substance.

DETECTION OF PHOSPHORUS.

The Presence of Phosphorus in an organic substance may be detected by converting it into PH_3, or into phosphate.

640. **Heating with Magnesium.**—Carbonise the substance by heat, then mix it with magnesium-powder and heat the mixture strongly: now moisten the cold residue with water: an onion-like smell of PH_3 will be observed, proving the production of magnesium phosphide, and the presence of phosphorus in the original substance.

641. **Ignition with $NaNO_3$, and Formation of Phosphate.**—Heat the solid substance with fused Na_2CO_3 and $NaNO_3$, as has been described for the detection of sulphur (639). Then dissolve the residue in dilute HNO_3, and add a few drops of this liquid to some $AmHMoO_4$-solution and warm gently: the formation of a yellow precipitate proves that phosphate has been formed, and indicates the presence of phosphorus in the original substance (578).

DETECTION OF CHLORINE, BROMINE, AND IODINE.

The Presence of the Halogen Elements in an organic substance may be detected either by the flame-coloration of their copper salts, or by converting them into sodium salts and then applying the ordinary tests to the solution of these salts.

642. Copper Flame-coloration.—Fix a piece of CuO in a loop of platinum wire, and heat it strongly in the outer Bunsen-flame until it gives no colour to the flame. Then dip the CuO into the organic substance (chloral) and ignite it again, first in the inner and then in the outer Bunsen-flame: a green flame coloration with a blue centre will appear, and will indicate the presence of chlorine in the organic substance. A similar result is obtained if the organic substance contains either bromine or iodine.

This test, therefore, only serves to indicate the presence of a halogen, and does not distinguish between the different halogen elements.

643. Ignition with Soda-lime or with Sodium.—Mix a little of the organic substance (chloral) with soda-lime free from chloride, place the mixture in a hard glass test-tube, and fill up the tube with soda-lime. Heat the soda-lime to redness (632, fig. 75), and then gradually extend the heating to the mixture. Dissolve the cold soda-lime in dilute nitric acid, and test the clear solution for chloride, bromide and iodide in the ordinary way (552, *et seq.*).

Or the substance may be ignited with a small piece of sodium, and the clear solution obtained by extracting the cold residue with water is then examined by 552, *et seq.* A cyanide will be formed if nitrogen was present in the substance (637, 647, *et seq.*).

II.—DETERMINATION OF SPECIFIC GRAVITY, MELTING-POINT, AND BOILING-POINT.

The Identification of a pure Organic Substance is frequently aided by the determination of its specific gravity and of its melting-point if solid, or its boiling-point if liquid, since these are constant properties. These determinations may take the place of analytical tests, but they are more frequently used with the object of confirming the results which are yielded by analytical reactions.

The determination of the specific gravity of a solid substance involves accurate weighing on a chemical balance. Directions for the process are given in Section I. in *Cloues and Coleman's Quantitative Analysis.*

The specific gravity of a liquid may be determined by accurate weighing, but the determination by means of a correct hydrometer (644) is much more rapid and convenient.

When a solid substance possesses a melting-point which can be taken by a mercurial thermometer, the process described in paragraph 645 may be employed, and will be found to be rapid and simple in execution. The fusing-points of most inorganic substances are, however, too high to admit of being determined by this process.

The determination of the boiling-point of a simple liquid, within the range of a mercurial thermometer, is described in paragraph 646 Mixed liquids may often be separated by fractional distillation (646a), and then identified by their boiling-points.

DETERMINATION OF THE SPECIFIC GRAVITY OF A LIQUID.

644. **The Hydrometer** serves to determine the specific gravity of a liquid rapidly, since it enables the process of weighing to be dispensed with. The apparatus consists of a glass or metal float (fig. 76), which is weighted below to such an extent as to cause it to assume a vertical position when it is floating in the liquid. The stem is so graduated that the numbered mark, which is level with the surface of the liquid, when the hydrometer is floating freely, shows the specific gravity of the liquid. The temperature of the liquid must be adjusted to the temperature at which the hydrometer has been graduated: this is usually 15·5° C.

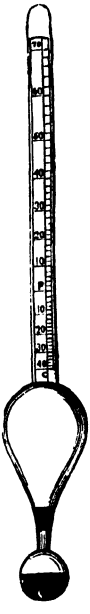
All specific gravities which are given in this book correspond to the temperature of 15·5° C.

DETERMINATION OF THE MELTING-POINT OF A SOLID.

645. **The Method commonly in Use** for determining the melting-point of a substance is illustrated in fig. 77.

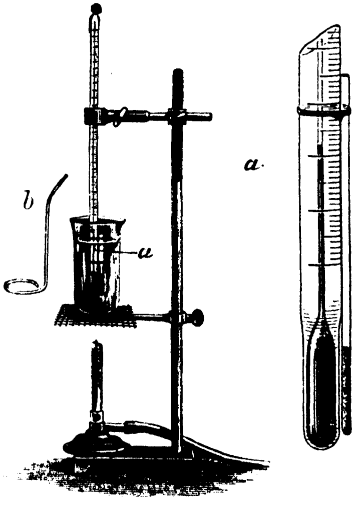
The solid substance is placed in a thin-walled glass tube, which is about $\frac{1}{16}$ inch in diameter and is sealed at one end.

FIG. 76.



HYDROMETER.

FIG. 77.



DETERMINATION OF MELTING-POINT.

Such a tube is readily made by drawing out, in the Bunsen-flame, a thin-walled narrow test-tube about $\frac{3}{16}$ inch in diameter.

This tube is attached to the stem of a delicate thermometer just above the bulb, by binding it by fine platinum wire. If the thermometer is to be dipped into water only, a little rubber ring, cut off some small rubber tubing, may be slipped over the larger end of the tube and the thermometer stem (fig. 77), instead of binding it with platinum wire.

The thermometer and tube are then placed in a beaker containing a liquid which has a higher boiling-point than the melting-point of the solid. Water, strong H_2SO_4, or melted paraffin-wax may be used in the beaker, according to the temperature which is required.

The beaker is then gently heated until the solid melts, and the temperature of liquefaction is accurately noted by the thermometer.

The process should be repeated several times with fresh portions of the substance, and the mean temperature, which has been obtained from these experiments, should be taken as the melting-point of the solid.

The following points require to be attended to.

If the mercury-thread of the thermometer extends above the surface of the liquid, the correction which is described in the *Note* in paragraph 646 must be applied to the reading.

During the process of heating, the contents of the beaker should be constantly mixed by means of a stirrer, which is made of bent glass rod or stout wire in the shape shown at *b* in fig. 77. This continuous stirring is necessary, in order to secure a uniform temperature throughout the mass of the liquid during the determination.

In the case of certain fats, and of some other substances, it is difficult to see the exact point of liquefaction. This is facilitated by using a tube which is open at both ends.

If there is any difficulty in introducing the substance into this open tube, the fine end of the tube may be inserted into the melted substance. A small quantity of the liquid will enter the tube by capillary attraction. This is allowed to cool and solidify, and the tube is then attached to the thermometer and immersed in the water as before.

At the moment of liquefaction, the substance will be forced up the tube by the pressure of the liquid in the bath. The temperature of the thermometer is noted when this movement is seen to occur.

It should be borne in mind that some substances, more especially the animal fats, show a lower melting-point when the process of fusion is repeated immediately after solidification. In such cases the normal melting-point is regained after the substance has remained in the solid condition for a sufficient interval of time.

EXPERIMENT.—The melting-point of naphthalene may be determined, using a beaker which contains water for the bath. The melting-point should be approximately 80° C.

DETERMINATION OF THE BOILING-POINT OF A LIQUID.

646. **The Boiling-point of a simple Liquid** may be determined in the following way.

The liquid is placed in a flask (fig. 78), the neck of which is closed by a perforated cork through which a thermometer passes, the bulb of the thermometer being close to the surface of the liquid. The liquid is then heated to boiling, and the bulb and part of the stem of the thermometer are thus surrounded by the vapour of the boiling liquid.

The neck of the flask has a side exit-tube, and if the liquid has a low boiling-point this side-tube should be connected with a cold-water condenser; but for less volatile liquids a plain glass tube, about thirty inches in length and cooled only by the atmosphere, will suffice (fig. 78).

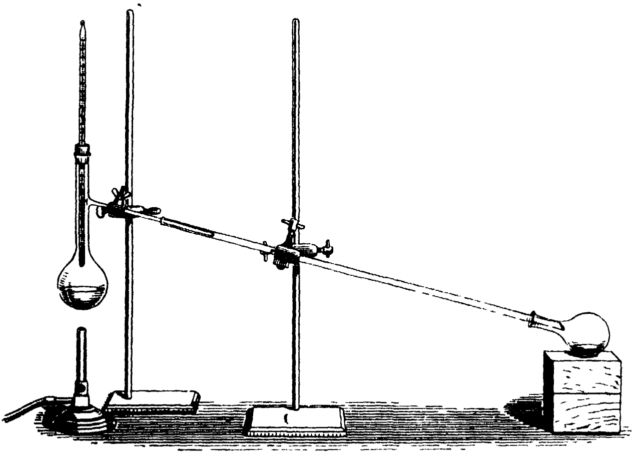
As soon as the reading of the thermometer remains constant, the temperature is registered as the boiling-point of the liquid.

Note.—This reading will, however, require to be corrected, if the thread of mercury in the thermometer extends above the cork. The following formula gives the correction to be added :—

$$N(T - t) \times 0{\cdot}000143.$$

Here N is the number of degrees on the thermometer-stem which are not heated by the vapour ; T is the temperature indicated by the thermometer ; *t* is the temperature taken by placing the bulb of a second thermometer midway between the cork and the top of the

FIG. 78.



DETERMINATION OF BOILING-POINT.

mercurial column during the process, the second bulb being screened from the direct heat of the Bunsen-flame ; and 0·000143 is the apparent coefficient of expansion of mercury.

It should be remembered that considerable differences of atmospheric pressure will cause appreciable variations in the boiling-point. All the boiling-points, which are given hereafter, correspond to the normal atmospheric pressure of 760 mm. of mercury.

EXPERIMENT.—The boiling-point of methylated spirit may be taken by this method. It should be approximately 78° C.

SEPARATION OF LIQUIDS BY FRACTIONAL DISTILLATION.

646a. Fractional Distillation may be employed to separate two or more miscible liquids which differ in their boiling-points by at least 20 degrees.

If a mixture of two such liquids is placed in a distillation flask (fig. 78, p. 254), fitted with a thermometer and a cold-water condenser, and the mixture is gradually heated, the following results will be observed.

The liquid will begin to boil at about the boiling-point of the more volatile liquid, but as the heating is continued the boiling-point will gradually rise. After a time the boiling-point will rise rapidly until it approaches the boiling-point of the less volatile liquid, when it will again rise slowly until the whole of the liquid has distilled.

If the volume of the distillate is measured from time to time, it will be found that the two largest volumes have been obtained while the thermometer indicates approximately the boiling-points of the two liquid constituents of the mixture; and a partial separation of the constituents has therefore been effected.

If each of these two main "fractions" is now separately distilled, it may be separated in a similar manner; and a fairly pure sample of each of the constituent liquids may be obtained by again collecting separately those fractions of the distillate which pass over while the thermometer registers the boiling-point of the particular constituent.

By repeating this fractional distillation a pure specimen of each of the original liquids may ultimately be obtained.

The following example will illustrate this method of separation by fractional distillation.

EXPERIMENT.—Make a mixture of equal volumes of pure alcohol and of water. Place 150 c.c. of this mixture in the

distillation flask fitted with a thermometer and a cold-water condenser (fig. 78, p. 254). Heat the liquid gently with a naked flame, and receive the fractions which pass over between 78°–85°, 85°–92°, 92°–98°, and 98°–100° separately in four clean dry flasks.

When the volumes of these fractions A, B, C, and D, are measured, fraction A will be about one-third of the total quantity, and may be proved to contain the greater part of the alcohol by its smell and by its ready inflammability. The fractions B and C are smaller in amount, and contain a mixture of alcohol and water, the proportion of alcohol being so small that the mixture is not inflammable. Fraction D will be found to consist almost entirely of water by its tastelessness and freedom from smell.

A further separation may be effected by "refractionating" the fractions already obtained, until eventually the fraction with the lowest boiling-point will be almost pure alcohol and that with the highest boiling-point almost pure water.

It will be obvious that a separation has thus been effected, and that special methods may now be applied for the identification of each of the liquids. This method of procedure is often applicable to liquid mixtures.

ANALYTICAL REACTIONS OF ORGANIC SUBSTANCES.

III.—ACID-RADICLES.

CYANIDE (Cy).—Use freshly-made solution of KCy.

The alkaline cyanides smell faintly of bitter almonds, owing to the evolution of HCy : this acid is intensely poisonous.

647. **$AgNO_3$**: white precipitate (AgCy).

This precipitate is most easily obtained by pouring a drop of the KCy-solution into the $AgNO_3$-solution.

Show with three separate portions of the precipitate in the liquid, that the precipitate is soluble in KCy-solution when it is added in excess, and in AmHO, but is insoluble in dilute HNO_3.

Filter off some of this precipitate, and wash it with hot water until HCl causes no milkiness with the last few drops of the washing water. Then dry it, scrape it from the filter into a porcelain crucible, and heat it strongly ; metallic silver will remain. If the cool residue is warmed with a little dilute HNO_3, it will dissolve, giving off reddish brown gas, and when a drop of HCl is added to this solution, a curdy precipitate (AgCl) will be produced.

This method of examining the precipitate which has been produced by $AgNO_3$ distinguishes AgCy from AgCl, AgBr, and AgI ; these are also precipitated by $AgNO_3$ and are insoluble in HNO_3 : but when they are dried and strongly heated, they fuse without undergoing decomposition. Hence, when HNO_3 is warmed with the cool mass after fusion, it cannot dissolve out Ag ; and when HCl is added to the HNO_3, it either gives no precipitate, or it causes only a slight milkiness, which is due to the precipitate not having been perfectly freed from soluble silver salt.

This process is somewhat tedious and need seldom be used, since cyanide is readily detected and distinguished by the following reactions.

648. **"Prussian Blue" test.**—$FeSO_4$-solution and Fe_2Cl_6-solution are added to solution of cyanide, and the liquid is

then made strongly alkaline with KHO, and boiled; it is then cooled and acidified with HCl: *Prussian blue* [$Fe_4'''(FeCy_6)_3$] will be formed, and will appear as a deep blue precipitate: or, if the quantity of cyanide originally present was small, a bluish-green solution of the Prussian blue will be obtained.

If no cyanide is present, the addition of HCl will produce an almost colourless and perfectly clear liquid.

649. **Formation of Sulphocyanide.**—If dilute H_2SO_4 is added to a cyanide in a test-tube fitted as is shown in figure 73 (p. 202), and then a small piece of marble is dropped in and the liquid is boiled, HCy will be carried over by the CO_2 gas. If the gas is allowed to bubble through some Am_2S-solution, AmCyS will be formed.

If the solution is now boiled in a porcelain dish, and acidified when cold with HCl in order to remove unchanged Am_2S, the addition of several drops of Fe_2Cl_6 will produce a red colour (662). The colour will be easily seen against the interior of the white dish. This result proves that HCy has passed into the Am_2S, and therefore that cyanide was present in the original substance.

A little of this red liquid should be added to some $HgCl_2$-solution. If the red colour is destroyed, it was certainly due to the presence of a sulphocyanide.

When HCl is added to the Am_2S in the above reaction, white S will separate. If the yellow liquid has been previously boiled in a porcelain dish until it becomes colourless, filling in distilled water to prevent evaporation to dryness, the separation of S on the addition of an acid is prevented. But this separation of S is frequently advantageous, since its whiteness helps to show up even a faint reddish tint in the liquid.

The marble is added in order to cause evolution of CO_2 gas: the escape of the gas lessens the risk of the Am_2S being sucked back during the reaction · it also helps to carry over the HCy into the Am_2S solution, and to prevent it from remaining behind in the test-tube.

650. **The Formation of Sulphocyanide** can often be more simply secured, by boiling the solution of the cyanide

in a porcelain dish with yellow Am_2S. The Am_2S must be added until the yellow colour of the mixture remains after the liquid has been boiled for a short time. If a dark-coloured precipitate is formed, this must be allowed to settle, or must be filtered off, before the yellow colour of the filtrate can be seen.

The whole of the liquid is then filtered, if necessary, and is acidified with HCl in the porcelain dish. Fe_2Cl_6 is then added in small quantity: a blood-red colour will be produced, which will not be removed when the liquid is heated or is mixed with a little dilute HCl. The colour will be at once destroyed, however, when a few drops of the liquid are added to some $HgCl_2$-solution.

Note.—The methods described in paragraphs 648, 649, and 650 serve as exceedingly delicate tests for cyanide. The methods in 648 and 649 are often less readily performed than that in 650, but they exceed it in delicacy The method in 650 is only replaced by that in 649 in cases where, after the substance has been boiled with an excess of Am_2S, the filtrate is dark coloured and therefore the red coloration would be invisible.

The reactions in 648, 649 are given by many complex cyanides (653-661).

651. **$HgCy_2$ and AgCy do not show the Reactions for Cyanide** by methods 647, 648, 649, but when they are boiled with Am_2S, according to method 650, AmCyS will remain in the filtrate from the black precipitate of sulphide, and will give the red coloration with Fe_2Cl_6.

When solid $HgCy_2$ is heated in a small tube closed at one end, it gives off cyanogen gas, which burns with a peach-blossom coloured flame when it is kindled.

652. **Hydrogen cyanide, Hydrocyanic acid or Prussic acid (HCy),** is a colourless, volatile, inflammable liquid which boils at 26° C. It readily mixes with water, and the vapour emitted from the acid or its solution smells like bitter almonds. The acid is *highly poisonous* when it is inhaled as vapour or swallowed as liquid. Hydrocyanic acid is easily detected by the tests which have been already given for a cyanide.

FERROCYANIDE ($FeCy_6$)$^{\text{iv}}$.—Use solution of $K_4FeCy_6.3H_2O$.

The abbreviated formula, $K_4\overline{Cfy}.3H_2O$, is sometimes used (131).

653. **Fe_2Cl_6**: dark blue precipitate of *Prussian blue* ($Fe_4\overline{Cfy}_3$): insoluble in HCl, soluble in $H_2C_2O_4$ to a dark blue liquid, and changed by KHO into brown $Fe_2H_6O_6$.

The solubility in $H_2\bar{O}$, and the insolubility in HCl, may be shown by pouring off portions of the liquid containing the precipitate, heating them with HCl and with $H_2\bar{O}$ respectively, then filtering and noting whether the filtrate is rendered blue by the solution of the precipitate.

654. **$FeSO_4$**: light blue precipitate ($Fe''K_2\overline{Cfy}$), which becomes darker in colour by oxidation on exposure to the air, or on the addition of Cl- or Br-water, or when it is warmed with HNO_3: it is insoluble in HCl.

655. **$CuSO_4$**: a chocolate-coloured precipitate ($Cu''_2\overline{Cfy}$), insoluble in $H\bar{A}$.

656. **$AgNO_3$**: white precipitate ($Ag_4\overline{Cfy}$), insoluble in HNO_3 *and in AmHO*. When the precipitate is heated with HNO_3, it changes to orange-red $Ag_3\overline{Cfy}$ which is *soluble in AmHO*.

FERRICYANIDE ($FeCy_6$)$'''$.—Use freshly-made solution of K_3FeCy_6.

This formula is sometimes doubled and written as $K_6(FeCy_6)_2$. It may be abbreviated to $K_3\overline{Cfy}$.

657. **Fe_2Cl_6**: dark green or brown coloration; no precipitate is formed, as may be seen when water is added to the liquid until it becomes transparent.

658. **$FeSO_4$**: dark blue precipitate of *Turnbull's Blue* ($Fe''_3\overline{Cfy}_2$), insoluble in acids: its blue colour is destroyed by KHO.

659. **$CuSO_4$**: yellowish precipitate.

660 **$AgNO_3$**: orange-coloured precipitate ($Ag_3\overline{Cfy}$), insoluble in HNO_3, *soluble in AmHO.* Frequently a white residue of $Ag_4\overline{Cfy}$ is left when AmHO is added; this proves that $K_4\overline{Cfy}$ was present in the $K_3\overline{Cfy}$-solution.

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661. Detection of Ferro- and Ferricyanide.—Ferro- and ferricyanide can be found and distinguished by their first three tests, or by the precipitate which has been given by $AgNO_3$ in the solution acidified with HNO_3 This precipitate will have a more or less decided orange-red colour if a ferricyanide is present. If the precipitate is separated from most of the liquid by filtration or decantation and is gently heated with AmHO, white $Ag_4\overline{Cfy}$ will be left if a ferrocyanide was present, and when the filtered ammoniacal liquid is acidified with HNO_3 orange-red $Ag_3\overline{Cfy}$ will be reprecipitated.

SULPHOCYANIDE (CyS).—Use solution of KCyS.

662. **Fe_2Cl_6**: blood-red coloration, but no precipitate: the colour is weakened but not destroyed by HCl: it disappears entirely when the liquid is dropped into $HgCl_2$-solution, and differs in this respect from the similar coloration which is produced by certain other organic acid-radicles.

FORMATE.—Use Sodium formate, $NaHCO_2$.

Formic acid and formates much resemble acetic acid and acetates. A solid alkaline formate fuses and chars and emits CO and CO_2 when it is strongly heated.

663. **Fe_2Cl_6** imparts a deep red colour to the solution of a formate; the coloration is destroyed by the addition of HCl. When the red liquid containing excess of formate is boiled the Fe is completely precipitated as basic ferric formate, and the liquid becomes colourless. This reaction is precisely similar to that for acetate (672).

664. **$AgNO_3$ or $Hg_2(NO_3)_2$**, when it is heated with solution of formate, in the absence of free acid or of alkali, yields with difficulty a precipitate of black Ag or of grey Hg.

665. **Silver Mirror.**—When solution of formate is gently heated with ammoniacal solution of $AgNO_3$, it gives no silver mirror (*Note*, 694). But free formic acid, or solution of formate which has been acidified with $H\bar{A}$, gives a silver mirror when it is heated with $AgNO_3$-solution.

666. **$HgCl_2$**, when it is mixed with solution of formate and heated to about 70° C., yields a white precipitate (Hg_2Cl_2) if HCl and alkaline chlorides are absent.

667. **Strong H_2SO_4**, when it is heated with a formate, evolves CO gas, which burns with a blue flame. No charring or blackening occurs, and no CO_2 is evolved as in the case of an oxalate (675).

If a formate is distilled with *dilute* H_2SO_4, a distillate is obtained which emits the pungent smell of formic acid.

668. **Formic acid (H_2CO_2)** is a colourless fuming liquid with a pungent smell; it boils at 100° C., and is readily miscible with water, with alcohol, and with ether. It is a powerful reducing reagent.

ACETATE ($C_2H_3O_2$) or ($\bar{A}$).—Use $Na\bar{A}.3H_2O$.

669. **Ignition.**—Many solid acetates, if they are strongly heated, blacken and evolve the vapour of acetone; this vapour possesses a peculiar smell, and burns with a bright flame when it is kindled.

670. **Strong H_2SO_4**, when it is heated with an acetate, evolves vapour of $H\bar{A}$. This vapour, if it is smelt in large quantity, is very pungent; in smaller quantity it smells like vinegar.

671. Formation of Ethyl acetate.—If an acetate is first mixed with a little alcohol, and then with strong H_2SO_4, a fragrant smell of ethyl acetate ($C_2H_5\bar{A}$) will be emitted when the mixture is warmed. The smell is most pronounced when the liquid is allowed to cool for a short time, and is then shaken up.

672. Fe_2Cl_6, if it is added in small quantity to solution of an acetate, causes *a deep red coloration.*

If the red liquid containing an excess of the acetate is boiled, the Fe is completely precipitated as light brown basic acetate. the liquid therefore becomes colourless.

The red colour of the solution is changed to yellow by the addition of a few drops of dilute HCl; it is distinguished in this way from the coloration which has been caused by sulphocyanide (662).

673. Cacodyl reaction.—This reaction must be carried out with due caution, since the vapour of cacodyl is *very poisonous.*

When a solid acetate is heated with a minute quantity of As_2O_3 a disgusting smell is produced, which is due to the evolution of the vapour of cacodyl, $As(CH_3)_2$.

674. Hydrogen acetate, or Acetic acid ($H.C_2H_3O_2$), is known as *glacial acetic acid.* At temperatures below 17° C it is a colourless crystalline substance, which readily melts. it boils unchanged at 118° C., giving off a very pungent inflammable vapour. This acid does not redden blue litmus test-paper until it is diluted. It is miscible with water, with alcohol, and with ether in all proportions.

OXALATE (C_2O_4).—Use $Am_2C_2O_4.2H_2O$.

675. Strong H_2SO_4, if it is heated with a solid oxalate, causes an effervescence, which is due to the evolution of a mixture of CO and CO_2:—

$$Am_2C_2O_4 + 3H_2SO_4 = CO + CO_2 + 2AmHSO_4 + H_2SO_4.H_2O.$$

The CO may be detected by burning with a blue flame when a light is applied, and the CO_2 is found by rendering lime-water milky (505, 1, 3, 4).

No blackening or charring is caused by strong H_2SO_4.

Oxalates differ in this respect from most other organic acids and their salts.

676. Solution of $CaCl_2$, of $CaSO_4$, or of CaH_2O_2, when it is added to solution of an oxalate, gives a white precipitate (CaC_2O_4), which is insoluble in AmHO and in $H\bar{A}$, but soluble in HCl and in HNO_3. Keep this precipitate.

677. Conversion into Carbonate by Ignition.—Filter off the precipitate of CaC_2O_4 (676), and pour upon part of it some dilute HCl; it will not effervesce. Dry the remainder of the precipitate, and heat it for a moment to dull redness on platinum foil; it will be converted *without blackening* into $CaCO_3$, and this will effervesce when dilute HCl is poured upon it.

This is a general test for oxalate of K, Na, Ba, Sr, or Ca; since each of these oxalates, when it is ignited, leaves a *carbonate* which effervesces with an acid. Many other organic salts of these metals undergo a precisely similar change when they are ignited, but the change of these salts is usually attended by charring.

678. Evolution of CO_2 by Oxidation.—When an oxalate, either in the solid state or in strong solution, is heated with MnO_2 and H_2SO_4, it evolves CO_2:—

$$Am_2C_2O_4 + MnO_2 + 3H_2SO_4 = 2CO_2 + MnSO_4 + 2AmHSO_4 + 2H_2O.$$

Note.—If the MnO_2, which is to be used in the test, is not known to be free from carbonate, it is best to add a little H_2SO_4 to the MnO_2 in a test-tube and then heat the liquid. If CO_2 is found in the tube the heating should be continued, and the CO_2 should be removed occasionally by sucking fresh air into the test-tube through a glass tube which is pushed down near to the surface of the liquid.

As soon as no CO_2 can be detected in the tube after it has been heated for a short time, the liquid being still distinctly acid, the oxalate is added and the mixture is again heated. CO_2 will be rapidly given off from the oxalate, and may be detected by the ordinary methods (505, 1, 3, 4).

679. $AgNO_3$ gives a white precipitate ($Ag_2C_2O_4$), which does not darken in colour when the liquid is heated.

679a. $KMnO_4$-solution acidified with dilute H_2SO_4 is decolorised in the cold by a soluble oxalate. (Diff. from other organic acids.)

680. **Hydrogen oxalate, or Oxalic acid** ($H_2C_2O_4\ 2H_2O$), crystallises in colourless rhombic crystals, which are soluble in water and in alcohol, and slightly soluble in ether. The acid melts at 100° C., and at 150° C. it partly sublimes unaltered evolving vapour which causes coughing, and is partly decomposed, $H_2C_2O_4 = H.COOH + CO_2$. It yields the reactions in pars. 675, 676, 678, and 679a. The crystals dissolve readily in water, producing an acid poisonous liquid.

SUCCINATE.—Use Ammonium succinate, $Am_2C_4H_4O_4$.

BENZOATE.—Use Ammonium benzoate, $AmC_7H_5O_2$.

681. Succinic acid and succinate frequently smell of amber, while benzoic acid and benzoates commonly emit an aromatic smell of gum benzoin.

Succinate and benzoate closely resemble one another in their analytical reactions ; the following differences are therefore important.

682. **Distinctive Properties.**—Succinic acid crystallises in monoclinic prisms, which melt at 182° C. : it is readily soluble in water.

Benzoic acid usually crystallises in lustrous flat plates, which melt at 121° C. It is only slightly soluble in cold water ; the acid is therefore partially precipitated from its solution in alcohol when water is added, and when strong HCl or any other strong acid is added to an aqueous solution of a benzoate.

683. **Ignition.**—When succinic acid and benzoic acid are heated they emit fumes, which are very irritating to the throat, and which cause coughing when they are inhaled. Benzoates, however, give off benzene when they are heated with soda-lime : the benzene is recognised by its smell and by burning with a smoky flame, and by the properties described in paragraphs 789, 790.

684 **$BaCl_2$-solution,** when it is added together with AmOH and much alcohol, precipitates succinates but not benzoates. A similar difference is shown with $CaCl_2$.

685. **Fe_2Cl_6** gives a *reddish-brown* precipitate with succinate, and a *pale brown or buff* precipitate with benzoate.

If the ferric benzoate precipitate is treated with strong HCl, benzoic acid separates in crystalline scales.

MALATE.—Use solution of Malic acid ($C_4H_6O_5$) which has been carefully neutralised by AmHO.

686. This acid is a colourless, crystalline, deliquescent substance, having a pleasant sour taste: it is readily soluble in water and in alcohol, but not in ether.

687. **$CaCl_2$** : the addition of $CaCl_2$, followed by boiling or by the addition of alcohol, produces a white precipitate in a neutral solution. This precipitate is soluble in AmHO. (Difference from citrate.)

688. **$Pb\bar{A}_2$** gives a white precipitate, which fuses and then dissolves when it is heated with sufficient water. If this precipitate is transferred to a piece of porcelain and heated, it swells up into a light puffy mass.

689. **$AgNO_3$** gives a white precipitate, which becomes grey when it is heated.

690. **Strong H_2SO_4**, when it is heated with a malate, evolves CO and CO_2, and the liquid gradually darkens.

TARTRATE ($C_4H_4O_6$) or ($\bar{T}$).—For a Neutral tartrate, use $KNa\bar{T}$; for an Acid tartrate, $H_2\bar{T}$ or $NaH\bar{T}$.

691. **Ignition.**—When a solid tartrate is heated strongly in a tube, it chars and gives off a smell of burnt sugar.

692. **Strong H_2SO_4**, when it is heated with a solid tartrate, causes rapid blackening and evolution of a mixture of CO, CO_2 and SO_2. Accordingly if the mixed gases are passed successively through $K_2Cr_2O_7$-solution and through lime-water, SO_2 and CO_2 will be detected (505, 519); the residual CO gas may then be kindled, and it will burn with the characteristic blue flame of CO.

693. $AgNO_3$ produces in the strong solution of a neutral tartrate a white curdy precipitate ($Ag_2\bar{T}$), which is soluble in AmHO and in HNO_3. Keep this precipitate.

694. Production of a Silver Mirror.—Allow some of the precipitate from the preceding reaction to settle in a test-tube, and decant as much of the clear liquid as possible.

Then pour *very dilute* AmHO drop by drop into the tube, until the precipitate, after it has been shaken up with the liquid, is nearly but *not quite* dissolved.

Now place the test-tube in some cold water in a beaker, and heat the water to about 60°. A brilliant *mirror-like film of Ag* will be deposited upon the interior of the tube.

Note.—If the original solution of the tartrate is dilute, the mirror is most easily obtained as follows. Very dilute AmOH is added to solution of $AgNO_3$ until the precipitate which forms at first is nearly redissolved. This reagent is added to the neutral tartrate solution. When the mixture is warmed, the mirror will be formed.

If the liquid is quickly boiled, the Ag will be deposited as a dull black precipitate, which does not adhere to the glass and is not characteristic.

695. $CaCl_2$ precipitates from the solution of a neutral tartrate white or crystalline $Ca\bar{T}$: this precipitate often separates only after the liquid has been shaken well, and has then been allowed to stand for a time. It is soluble in acids, and its formation is retarded by the presence of AmCl or of an Am-salt.

When this precipitate is freshly formed, it is completely soluble in strong cold KHO solution which is free from K_2CO_3. If this alkaline solution of the tartrate is boiled, the $Ca\bar{T}$ is reprecipitated; but it dissolves again, more or less completely, when the liquid is cold.

Decant the clear liquid from the $Ca\bar{T}$-precipitate, and pour upon the precipitate a little very dilute AmHO. Then drop in a small crystal of $AgNO_3$ and heat slowly. A silver mirror will form on the part of the glass on which the crystal of $AgNO_3$ rests.

696. **KCl, or better KA,** if it is added to the solution of an acid tartrate, or to solution of a neutral tartrate which has been acidified with $H\bar{A}$, gives a white crystalline precipitate ($KH\bar{T}$); this precipitate forms slowly unless the liquid is vigorously stirred or shaken.

The precipitate is soluble in caustic alkalies and in mineral acids, but is almost insoluble in alcohol; hence alcohol is often added to hasten the precipitation.

697. **Fe_2Cl_6-solution,** if it is added in small quantity to solution of a tartrate, undergoes no precipitation when the liquid is made strongly alkaline by the addition of KHO or AmOH.

The precipitation of several other metals (such as Al, Mn and Cu) from their solutions by alkaline hydrates, is similarly prevented by the presence of a tartrate. This is owing to the formation of a double tartrate, which is not decomposed by the alkali.

698. **Hydrogen peroxide.** If a few drops of the peroxide are added to a tartrate solution, then some $FeSO_4$-solution and excess of KOH, a *violet coloration* will be produced. (Difference from citrate.) Instead of adding H_2O_2, NaOCl solution may be added to the tartrate-solution which has been previously acidified with $H\bar{A}$.

698a. **Resorcinol** and solid tartrate, when gently heated with a little strong H_2SO_4, yield a dark red coloration.

Tartar emetic answers to this test, but not to some of those which are given above.

699. **Hydrogen tartrate, or Tartaric acid** ($H_2\bar{T}$), forms colourless rhombic crystals, which are not altered by exposure to the air. It melts at 170° C., has a pleasantly sour taste, and is soluble in water and in alcohol, but not in ether. It is detected by being heated either alone or with strong H_2SO_4 (691, 692, or 698a); and its solution, if neutralised, yields the reactions in paragraphs 693–698.

CITRATE.—Use Sodium citrate, $Na_3C_6H_5O_7.2H_2O$.

700. A citrate much resembles a tartrate and an oxalate in its reactions; it differs by emitting pungent acid fumes when it is carbonised by heat, and by darkening when it is heated with strong H_2SO_4 only after the mixture has been heated for some time and inflammable gases have escaped.

Citrate differs from tartrate by giving no precipitate with $K\bar{A}$-solution (696), and by yielding no violet colour in the reaction in paragraph 698.

701. **$CaCl_2$, or Lime-water.**—Calcium citrate is only precipitated *after long standing, or when the liquid is boiled,* after the addition of either a large excess of lime-water, or of a mixture of AmHO, AmCl and $CaCl_2$. This precipitate is insoluble in KHO. (Difference from $Ca\bar{T}$.)

702. **$AgNO_3$** gives a white precipitate, which becomes grey when it is heated. Solution of citrate, if it is gently heated with ammoniacal $AgNO_3$-solution, causes either very slight precipitation of Ag or none at all (694). (Difference from tartrate.)

703. **Hydrogen citrate, or Citric acid ($H_3C_6H_5O_7.H_2O$),** crystallises in transparent rhombic prisms. It is very soluble in water, less soluble in alcohol, and only slightly soluble in ether. It melts at 100° C. losing water and forming aconitic acid, and finally blackens giving off irritating fumes.

SALICYLATE.—Use Salicylic acid, $C_7H_6O_3$.

704. **Ignition.**—Salicylic acid melts at 157°, and at a somewhat higher temperature it decomposes into CO_2 and phenol or carbolic acid (C_6H_6O). It is sparingly soluble in cold water, but readily soluble in hot water.

705. **Ignition with Soda-lime.**—The decomposition of salicylic acid by heat is rapid and complete, if the acid has been mixed with excess of soda-lime before it is heated; phenol condenses as a white crystalline sublimate in the cooler part of the tube.

706 **Fe_2Cl_6.**—The solution of salicylic acid becomes *violet* when Fe_2Cl_6-solution is added. This colour is destroyed by HCl but not by $H\bar{A}$. (Difference from phenol.)

707. **Heated with H_2SO_4 and Methyl alcohol,** salicylate gives a fragrant smell of methyl salicylate (oil of wintergreen).

708. **H_2SO_4 and Nitrite.**—The addition to salicylate of strong H_2SO_4, which has been mixed with a small quantity of 6 per cent. aqueous solution of KNO_2, causes a permanent red coloration. (Difference from phenol.)

TANNATE, GALLATE.—Use Tannic Acid, $C_{14}H_{10}O_9$, and Gallic acid, $C_7H_6O_5$.

709. These acids and their salts resemble one another in many analytical reactions. Tannic acid is fairly soluble in cold water, but gallic acid is only slightly soluble. The following differences are important.

710. **Fe_2Cl_6** yields a *bluish-black* precipitate with each of these acids. The precipitate which has been caused by gallic acid disappears when the liquid is boiled.

711. **$CuSO_4$,** in ammoniacal solution, gives a greenish-coloured precipitate at once with a tannate.

With a gallate either the precipitate is not produced at all, or it forms only after a time.

712. **KCN,** if it is freshly dissolved, gives a red coloration

with a gallate; the colour gradually fades, but it reappears when the liquid is shaken.

No colour is produced when KCy-solution is added to a tannate.

713. **Strong H_2SO_4**, when it is heated with a tannate, gives a brown coloration; when it is heated with gallate a rich red coloration is produced which changes to claret colour. In each case SO_2 is eventually evolved.

714. **Gelatine** is precipitated from its solution by tannic acid. Gallic acid does not precipitate gelatine.

Meconate.—Use solution of Meconic acid, $C_7H_4O_7.3H_2O$, neutralising it with dilute AmOH.

715. Meconic acid is present in opium and in opium extracts It forms colourless crystalline scales, which are slightly soluble in cold water, and readily soluble in hot water and in alcohol.

716. **Fe_2Cl_6.**—Meconic acid may be detected by the production of a *deep red* coloration when it is mixed with Fe_2Cl_6; this colour is with difficulty destroyed by HCl.

This coloration also differs from that caused by an acetate or by a formate, by not disappearing when the liquid is boiled: it may be distinguished from ferric sulphocyanide by not being destroyed when the liquid is dropped into solution of $HgCl_2$.

717. **$Pb\bar{A}_2$-solution** gives in solution of a meconate a white precipitate of lead meconate.

718. **$CaCl_2$-solution** gives in neutral solution of a meconate a white precipitate [$Ca_3(C_7HO_7)_2$].

719. **$AgNO_3$** gives in strong solution of a meconate a white precipitate of $Ag_2H_2C_7O_7$, which changes to bright yellow $Ag_3HC_7O_7$ when a drop of AmHO is added.

URATE.—Use Potassium urate, $KC_5H_3N_4O_3$.

720. Uric acid is a white crystalline powder, which is almost insoluble in hot and cold water.

721. **HNO_3** dissolves the acid with effervescence : if this solution is evaporated to dryness in a porcelain dish, and the residue is then moistened with AmHO unless AmHO was originally present, bright red *murexide* is formed ; this becomes purple when KHO is added.

722. **HCl** precipitates white uric acid from solution of a urate.

723. **Ignition**.—When a urate is heated in an ignition-tube it chars and gives off NH_3

HIPPURATE.—Dissolve Hippuric acid, $C_9H_9NO_3$, in very dilute AmOH-solution, and neutralise if necessary.

724. This acid occurs in colourless prismatic crystals : it has a bitter taste and a strongly acid reaction : it is only slightly soluble in cold water, but is freely soluble in hot water and in alcohol.

725. **HCl** causes a white crystalline precipitate of hippuric acid in a neutral solution of a hippurate.

726. **Fe_2Cl_6** causes a flesh-coloured precipitate : this is decomposed by HCl, with separation of crystalline needles of hippuric acid.

727. **$AgNO_3$** gives a white precipitate.

728. **Ignition with Soda-lime.**—When a solid hippurate is heated with *soda-lime*, it evolves NH_3 and benzene (C_6H_6).

PICRATE, PICRIC ACID OR TRINITRO-PHENOL, $C_6H_3(NO_2)_3O$.

729. **Picric acid** is a yellow crystalline substance, which melts at 122·5° C. It is intensely bitter and stains the skin yellow. It is very slightly soluble in cold water, but is soluble in hot water, in alcohol, and in ether. The metallic picrates are very explosive.

730. **Ignition.**—When picric acid is heated in an ignition-tube it fuses, and then explodes slightly giving off dense black fumes.

731. **Bleaching-powder** : when picric acid is heated with bleaching-

powder-solution, it gives off a pungent odour resembling that of mustard-oil; this is due to the formation of chlorpicrin.

732. **KHO and Glucose,** when they are heated with picric acid, yield a brown colour this is due to the formation of picramic acid.

733. **Ammonia-copper sulphate solution,** made alkaline with AmHO, gives a bright green precipitate.

IV.—ALKALOIDS.

734. **Most Alkaloids are basic substances,** and resemble NH_3 in their behaviour with acids. Their solutions are alkaline to test-paper; the solutions of their salts are either neutral or acid in reaction. The free alkaloids are almost insoluble in water, hence solutions of their salts must be employed for trying the liquid reactions: the following reactions are generally given by these solutions.

735. **Na_2CO_3** precipitates the hydrated alkaloid from strong solutions of its salts.

736. **Phospho-molybdic acid** causes a yellow precipitate in the solution of an alkaloid

Note.—This reagent is prepared as follows:—The solution of ammonium molybdate in nitric acid (1162, 72) is precipitated by the careful addition of sodium phosphate solution (1160, 14). The precipitate is well washed, and is then dissolved in Na_2CO_3-solution. This solution is evaporated to dryness and the residue is ignited. Water is then added and warmed with the residue, which is finally dissolved by adding considerable excess of HNO_3.

736a. **$PtCl_4$**-solution, acidified with HCl, gives in solution of an alkaloid salt a yellow crystalline precipitate, similar to that obtained in solution of an ammonium salt.

737. **Iodine dissolved in KI-solution** gives a reddish-brown precipitate in the solution of an alkaloid.

738. **Ignition.**—When an alkaloid is heated on platinum foil it burns with a bright smoky flame: and when it is heated with soda-lime it evolves NH_3.

QUININE.—Use Quinine sulphate, $C_{20}H_{24}N_2O_2.H_2SO_4.8H_2O$.

CINCHONINE.—Use Cinchonine sulphate:—
$C_{19}H_{22}N_2O.H_2SO_4.2H_2O$.

739. **HCl and Ignition.**—Both quinine and cinchonine, when they are mixed with HCl and evaporated to dryness, leave residues, which, when they are ignited, evolve purple vapour similar in appearance to iodine vapour.

740. **Fluorescence.**—When quinine sulphate is being dissolved in water, a few drops of dilute H_2SO_4 should be added; the solution is intensely bitter, and gives a pale blue fluorescence.

The similar solution of pure cinchonine sulphate is not fluorescent.

741. **Alkaline hydroxide or carbonate** precipitates hydrated quinine from fairly strong solutions of a quinine salt; this precipitate disappears when the liquid is shaken with ether.

The corresponding cinchonine precipitate is not dissolved by ether.

742. **Br-water, or Cl-water,** when it is added in small quantity, does not colour a solution of quinine: but if AmHO is afterwards added, an intense *emerald-green* colour appears.

Cinchonine-solution under these conditions yields a yellowish-white precipitate.

743. **Br- or Cl-water and K_4FeCy_6.**—If the addition of Br- or Cl-water to quinine solution is followed by the addition of K_4FeCy_6-solution and one or two drops of KHO-solution, a *deep red* tint is produced. This colour quickly changes to dirty brown: it is destroyed by $H\bar{A}$, but reappears when AmHO is cautiously added.

744. **K_4FeCy_6** gives a yellow precipitate in cinchonine solution: the precipitate is easily soluble in excess: it is also soluble in hot water, and crystallises from this solution as it cools.

MORPHINE.—Use Morphine hydrochlorate,
$C_{17}H_{19}NO_3.HCl.3H_2O$.

745. This alkaloid is present in opium and in opium extracts: it is prepared from opium.

746. **KHO or AmHO** yields a white precipitate, which is soluble in excess of the reagent.

747. **HNO_3**.—The strong acid produces a *yellowish-red* colour, which does not change to *violet* on the addition of $SnCl_2$. (Difference from brucine.)

748. **Fe_2Cl_6-solution**: if the neutral solution is added drop by drop to neutral morphine solution, it produces a *dark blue* colour: this colour dissappears when an acid is added.

749. **Iodic acid**, when it is added to morphine and its salts, yields free iodine. The separation of iodine is shown by the liquid becoming brown; but the iodine is more certainly and readily detected by adding starch-solution, or by shaking the liquid with CS_2 (565, 2, 3). The brown coloration of the solution becomes more intense on the addition of AmHO.

This test is very delicate and characteristic, if the solid substance is moistened with a solution of one part of iodic acid in fifteen of water, and solution of one part of starch in four hundred of water is then added; when very dilute AmHO is poured upon the blue solution thus obtained, a coloured ring is seen at the surface of contact of the two liquids; the ring is *blue* below and *brown* above.

This reaction serves to distinguish morphine from other organic substances which contain nitrogen.

750. **H_2SO_4** gives no colour, when it is added to a solution containing morphine; but when a crystal of $K_2Cr_2O_7$ is dropped into the acid liquid contained in a white porcelain dish, and the crystal is slowly moved about with a glass rod, an *intense green* colour appears. Quinine gives a similar reaction.

751. H_2SO_4 and $K_2Cr_2O_7$.—If solid morphine or any of its compounds is dissolved by heating it with a few drops of strong H_2SO_4, and a minute quantity of HNO_3 is added to the cold solution, a deep red colour is produced. This colour turns to a mahogany tint when a fragment of $K_2Cr_2O_7$ is dropped into the liquid.

752. $AmHMoO_4$: solution of the molybdate in strong H_2SO_4 produces an intense purple coloration, which changes to blue.

STRYCHNINE.—Use Strychnine, $C_{21}H_{22}N_2O_2$.

753. When strychnine is being dissolved in water, a drop of dilute H_2SO_4 should be added. The solutions of strychnine are very poisonous: they possess an intensely bitter taste even when they are extremely dilute.

754. H_2SO_4 and $K_2Cr_2O_7$.—Strychnine dissolves in strong H_2SO_4 to a colourless liquid. When a fragment of $K_2Cr_2O_7$ is added to this solution in a porcelain dish, and is slowly moved about by pushing it with a glass rod, a *bluish-violet* coloration is produced which gradually changes to red or yellow.

The presence of morphine, or of a metallic chloride or nitrate, interferes with this reaction. If any of these substances are present, the strychnine may be precipitated by the addition of solution of K_3FeCy_6, or of K_2CrO_4; the precipitate is filtered off and slightly washed on the filter, and is then stirred with strong H_2SO_4.

755. HNO_3.—The cold strong acid dissolves strychnine without becoming coloured, but the solution acquires a yellow tint when it is heated.

NARCOTINE.—Use Narcotine, $C_{22}H_{23}NO_7$, or its sulphate.

756. Narcotine is present in opium, but it is not extracted from opium by the processes usually employed for obtaining morphine.

757. **H_2SO_4** : the strong acid gives a *bluish-violet* coloration, which changes to orange ; with some specimens a yellow solution is produced at once.

When this liquid is *gradually warmed*, it becomes first *orange-red* and then *bluish-violet*, or *purple* stripes proceed from the edge of the liquid surface: when the liquid is cooled, the colour changes to *cherry-red*. If the heating is pushed to the initial evaporation of the acid, an intense *reddish-violet* colour is produced.

758. **H_2SO_4 and $K_2Cr_2O_7$** —If narcotine is subjected to the reaction which has been already described for morphine in paragraph 751, an intense red coloration is produced.

759. **HNO_3** : the strong acid produces a yellow coloration in the cold liquid : this changes to red when the liquid is heated, but fades to yellow again as the liquid cools.

BRUCINE.—Use Brucine, $C_{23}H_{26}N_2O_4.4H_2O$, or its sulphate.

This substance may be dissolved in water to which a drop of H_2SO_4 has been added

780. **HNO_3** : the strong acid yields an *intensely red* coloration, which gradually changes to yellowish-red and yellow when the liquid is heated. When $SnCl_2$ is added to this hot solution, it assumes an intense *violet* colour. (Difference from morphine.)

781. **H_2SO_4** : the strong acid produces a *rose-coloured* solution, which changes to *yellow*.

782. **H_2SO_4 and $K_2Cr_2O_7$** : if the reaction which has already been described for morphine (751) is carried out with brucine, the liquid becomes red but quickly changes to yellow.

CAFFEINE, OR THEINE, $C_8H_{10}N_4O_2.H_2O$.

783. Caffeine is a colourless substance which forms needle-like crystals. It melts at 225° C, and sublimes unchanged. It is slightly soluble in cold water, and readily soluble in hot water. It is fairly soluble in alcohol and in ether.

784. **Formation of Murexide.**—If even a *trace* of caffeine is mixed with strong HNO_3 and then evaporated, a yellow residue remains: this assumes an intense violet colour when AmHO is poured upon it.

785. **Chlorine-water.**—If chlorine-water is added and the mixture is evaporated, a brown residue is obtained. this residue dissolves in AmHO and yields a violet-red solution.

V.—HYDROCARBONS.

Note.—All boiling-points are assumed to be taken under the normal atmospheric pressure of 760 millimetres, and all specific gravities correspond to a temperature of 15 5° C.

TURPENTINE, $C_{10}H_{16}$.

786. **Turpentine** is a liquid which possesses a characteristic smell, and boils at 156° (646); its specific gravity is 0·876 (644). When it has been warmed it is easily kindled, and burns with a very smoky flame. It is not miscible with water, but readily dissolves in strong alcohol and in ether.

787. **Strong H_2SO_4**: when this acid is added in small quantity to turpentine and the mixture is warmed, a peculiar and very characteristic smell is evolved.

788. **Br-water**, when it is shaken up with turpentine, is at once decolorised.

BENZENE, OR BENZOL, C_6H_6.

789. **Pure Benzene** is a very mobile, volatile, strongly smelling liquid, which burns readily with a smoky flame. It boils at 80° C., and its specific gravity is 0·8839. It is miscible with alcohol and with ether, but is insoluble in water.

790. **HNO_3**: if the strong fuming acid, or a mixture of strong H_2SO_4 and strong HNO_3, is warmed with benzene, nitro-benzene ($C_6H_5NO_2$) is produced: this substance separates as a reddish-yellow oil, smelling of bitter almonds, when the acid liquid is largely diluted with water.

If this nitro-benzene is poured into dilute HCl and pieces of zinc are introduced, aniline is formed and may be detected by the tests in paragraphs 847–851.

VI.—HALOID COMPOUNDS.

CHLOROFORM, $CHCl_3$.

791. **Pure chloroform** is a colourless, mobile liquid, with an ethereal smell. It boils at 61·4°, and has a specific gravity of 1·5. It is only slightly soluble in water, but is miscible with alcohol and with ether.

792. **Formation of Phenyl isocyanide.**—When chloroform is mixed with a drop of aniline, and with some solution of KHO in alcohol, and the mixture is heated, an *intensely disagreeable* smell of phenyl isocyanide is given off.

793. **Fehling's-solution** (864), when it is warmed with chloroform, deposits red cuprous oxide after some time.

794. **Phenol and KHO.**—If a mixture of alcoholic solution

of phenol with KHO is evaporated to dryness on the water-bath, the residue assumes a beautiful purple colour when it is moistened with chloroform. (Difference from chloral.)

IODOFORM, CHI_3.

795. Iodoform is a bright yellow substance, which emits a characteristic smell, and crystallises in hexagonal plates or stars. It melts at about 117° (645), and then vaporises, evolving iodine and HI and leaving a residue of carbon. It is insoluble in water and in acids, but dissolves in alcohol and in ether. When it is boiled with KOH-solution it breaks up into a mixture of potassium formate and potassium iodide, which may be separately identified.

VII.—CERTAIN ALCOHOLS AND PHENOLS.

METHYL ALCOHOL, CH_4O.

796. **Pure methyl alcohol** resembles pure ethyl alcohol (800) in its general properties; it boils at 66° C., and has a specific gravity of 0·7972. It burns with a pale blue flame and is miscible with water and with ether in all proportions.

797. **Oxidation to Formic acid.**—If a mixture of methyl alcohol with dilute H_2SO_4 and $K_2Cr_2O_7$ is distilled (83, 646a), the distillate will contain formic acid: when this *acid distillate* is warmed with $AgNO_3$, it will cause a brilliant silver mirror to be deposited.

When ethyl alcohol is oxidised by similar treatment, it yields aldehyde; and this only deposits a silver mirror when it is warmed with $AgNO_3$ *in an alkaline solution.*

798. **Formation of Methyl salicylate.**—When methyl alcohol is heated with strong H_2SO_4 and salicylic acid, a fragrant smell of methyl salicylate, or oil of winter-green, is evolved.

799. Iodoform is not produced when pure methyl alcohol is warmed with excess of iodine in the presence of Na_2CO_3 (**801**). (Difference from ethyl alcohol.)

ALCOHOL, OR ETHYL ALCOHOL, C_2H_6O.—Use rectified spirit diluted with four times its volume of water.

800. Pure ethyl alcohol is a mobile fragrant liquid: it boils at 78° (**646**), has a specific gravity of 0·7938 at 15·5° (**644**), and burns with a pale blue smokeless flame. It is miscible in all proportions with water and with ether.

If much water is present with the alcohol, it may be necessary to add dry K_2CO_3 in excess to the liquid, and then to distil over about one-third of it (**83**, **646a**). The alcohol is thus concentrated in the distillate: and it is much more readily detected by the following tests in the distillate than in the original dilute liquid.

801. Formation of Iodoform.—If Na_2CO_3-solution is added to water which contains a little alcohol, and the mixture is gently heated for some time, small pieces of iodine being occasionally added until the solution retains a brown colour, *golden yellow crystals of iodoform* will separate. If very little alcohol was present, the liquid may require to stand for ten or twelve hours before the crystals make their appearance.

When these crystals are examined under the microscope, they are seen to be hexagonal tablets or six-pointed stars.

This reaction is yielded by other substances besides ethyl alcohol, and is therefore not altogether characteristic

802. Formation of Aldehyde and Acetic acid.—Alcohol may be converted by oxidation into aldehyde and acetic acid by heating it for some time with $K_2Cr_2O_7$ and H_2SO_4. During the process of oxidation, aldehyde will be first smelt, and then acetic acid. If the smell is not distinctly recognised, the liquid may be distilled and the first portion of the distillate divided into two portions: one portion may then be tested for aldehyde by paragraph 823, and the other may be neutralised with Na_2CO_3, evaporated to dryness, and the solid residue tested for acetate (670–672).

Acetic acid may result from the oxidation of other substances besides alcohol.

803. **Benzoic- and Acetic-ether.**—When ethyl alcohol is heated with strong H_2SO_4 and a benzoate or an acetate, the characteristic smell of benzoic ether or of acetic ether may be recognised.

GLYCEROL, OR GLYCERINE, $C_3H_8O_3$

804. **Pure glycerol** is a colourless, viscous liquid, with intensely sweet taste. It has a specific gravity of 1·27, a melting-point of 20° C., and a boiling-point of 290°. It burns with a somewhat luminous flame, and is miscible with water and with alcohol, but not with ether.

805. **$NaHSO_4$.**—When glycerine is heated with $NaHSO_4$ it evolves intensely pungent acrolein vapour. If this vapour is passed into solution of rosaniline, which has been previously decolorised by SO_2, it produces a red coloration: other aldehydes produce the same result.

806. **Phenol and H_2SO_4.**—If phenol, strong sulphuric acid and glycerine are mixed together in equal volumes, and the mixture is heated to 120° C., and is then diluted and mixed with excess of AmOH, it becomes *crimson* in colour.

PHENOL, OR CARBOLIC ACID, C_6H_6O.

807. **Pure Phenol** is a colourless crystalline substance; it is, however, commonly tinged with pink. Its smell is very characteristic. It melts at 41° C., and boils at 182° C. Its specific gravity is 1·0702. It is slightly soluble in water, freely soluble in benzene, in alcohol, in ether, and in caustic

alkali solution, but insoluble in solution of alkaline carbonate.

808 **Fe_2Cl_6-solution** gives with phenol a violet coloration, which is destroyed by $H\bar{A}$.

809. **Bromine-water,** or **Bleaching-powder,** when it is added to phenol solution, which has been mixed with a quarter of its volume of dilute AmHO, produces a blue coloration : this colour changes to red on the addition of an acid.

810 **Nitrous acid.**—When a mixture of strong H_2SO_4 with a little 6 per cent. solution of KNO_2 is added to phenol, it produces a reddish-brown coloration, which changes to green and purple, and finally to blue.

811. **Bromine-water,** when it is added to phenol solution, gives a pale yellow precipitate of tribromphenol bromide.

HYDROQUINONE, $C_6H_6O_2$.

812. Hydroquinone is a white crystalline substance which shows a greenish tint. It melts at 169° C., and is soluble in water, in alcohol, and in ether.

813. **Ferric chloride,** if it is added to a dilute solution of hydroquinone, gives a green coloration, which rapidly changes to reddish-brown. If it is added to a strong solution, or to solid hydroquinone, a dark green crystalline precipitate is formed.

814. **$AgNO_3$**-solution is reduced by hydroquinone, and metallic Ag is precipitated.

815. **KHO**-solution becomes brown when it is shaken with hydroquinone solution.

815a. **$KMnO_4$**-solution acidified with dilute H_2SO_4, when boiled with hydroquinone evolves the irritating odour of quinone.

PYROGALLOL, OR PYROGALLIC ACID, $C_6H_9O_3$.

816. Pyrogallol is a white crystalline substance : it melts at 115° C. and is readily soluble in water, in alcohol and in ether.

817. **KHO** and other alkalis readily dissolve this substance ; the solution rapidly becomes brown by exposure to the air, owing to the absorption of oxygen.

818. **$FeSO_4$** colours pyrogallol solution dark blue

819. **Fe_2Cl_6** gives a fine red coloration to pyrogallol solution.

820. **$AgNO_3$** is immediately reduced by pyrogallol with the precipitation of Ag.

VIII.—ALDEHYDES AND KETONES.

ALDEHYDE, OR ACETALDEHYDE, C_2H_4O.

821. **Pure acetaldehyde** is a very mobile liquid, possessing a most characteristic smell: it boils at 21° C., and has a specific gravity of 0·7876 unless it is partially polymerised. It is miscible with water, with alcohol and with ether in all proportions.

822. **$AgNO_3$**: if $AgNO_3$-solution is mixed with very dilute AmHO until the precipitate, which forms at first, just disappears, and the solution is then heated with aldehyde, a silver mirror is deposited on the glass. (Proceed as in 694.)

823. **Magenta**-solution, which has been decolorised by SO_2, produces a violet-red colour when it is mixed with aldehyde.

824. **KHO**-solution, when it is heated with aldehyde, produces aldehyde-resin, a yellow substance with a peculiar smell.

825. **H_2S**, when it is passed into the aqueous solution of aldehyde, produces an oily liquid, which is changed by acids into a solid polymer.

826. **$NaHSO_3$** in saturated solution, gives a crystalline precipitate ($C_2H_4O.NaHSO_3$), which is decomposed by acids and by alkalis with liberation of aldehyde.

CHLORAL.—Use Chloral hydrate, $C_2HCl_3O.H_2O$.

827. Pure chloral (C_2HCl_3O) is a colourless liquid, with a pungent tear-exciting smell: it boils at 98°, and has a specific gravity of 1·4. It unites with water, forming a crystalline hydrate, which melts at 57° and boils at 97·5°, and has a specific gravity of 0·848.

828. **Phenyl isocyanide test.**—When chloral is heated with alcoholic solution of KHO and aniline, it yields phenyl isocyanide, which is recognised by its extremely offensive smell. (Refer to 792.)

829. **Silver mirror.**—When chloral is warmed with ammoniacal $AgNO_3$-solution (822) and a drop of KHO solution, a silver mirror is readily deposited on the interior of the glass vessel. (Difference from chloroform.)

Chloral also reduces Fehling's solution (864), yielding red Cu_2O.

830. Chloral gives the magenta reaction (823).

831. **KOH.**—Chloral is decomposed by aqueous KHO solution: chloroform separates in minute drops, and potassium formate remains in solution.

BENZALDEHYDE, OR OIL OF BITTER ALMONDS, C_7H_6O.

832. This liquid is colourless and highly refractive: it has a specific gravity of 1·05, and boils at 179° C.: it is sparingly soluble in water, and readily soluble in alcohol and in ether. On exposure to the air it is slowly converted into benzoic acid (682, 683).

833. **KHO** · when benzaldehyde is heated with solid KHO, benzy alcohol and potassium benzoate are formed. If the resulting solution is acidified with HCl, benzoic acid will be precipitated.

834. **$AgNO_3$**, in ammoniacal solution, is reduced by this aldehyde to metallic Ag.

835. **Na_2SO_3-solution** dissolves benzaldehyde: the careful addition of dilute H_2SO_4 to this solution precipitates crystalline flocks of $C_7H_6O.NaHSO_3$.

ACETONE, C_3H_6O.

836. **Pure acetone** is a mobile liquid, with a peculiar and characteristic smell: it boils at 55.6° C., and has a specific gravity of 0·7965. It is miscible with water, with alcohol and with ether in all proportions.

837. **Iodine**: if iodine dissolved in AmI-solution is added to dilute AmOH, it causes a black precipitate ($NH_3\,NI_3$); this precipitate disappears at first when the liquid is shaken, but ultimately tends to become permanent. If acetone is present in the dilute ammonia solution, iodoform separates, and this is characterised by its crystalline appearance under the microscope (**801**). Acetone is thus detectable in alcohol.

838. **$HgCl_2$**-solution, if it is made strongly alkaline by alcoholic solution of KOH, yields a precipitate of HgO: when the liquid containing the precipitate is shaken with acetone, part of the HgO is dissolved; if the liquid is filtered, the presence of Hg may be shown in the filtrate either by adding Am_2S (**279**), or by acidifying the liquid with HCl and adding $SnCl_2$ (**280**).

839. **Sodium nitro-prusside**, if it is added to acetone solution which has been previously mixed with twice its volume of strong NaOH-solution, produces a *bright red* coloration: this coloration becomes bluish when $H\bar{A}$ is added.

840. **$NaHSO_3$**, in saturated solution, gives a crystalline precipitate ($C_3H_6O.NaHSO_3$), which is decomposed by acid or alkali with liberation of acetone.

840a. **Parabromphenyl hydrazine**, dissolved in glacial acetic acid, then diluted and mixed with acetone, gradually yields yellowish needles of bromphenyl hydrazone.

IX.—AMIDO-COMPOUNDS.

UREA, OR CARBAMIDE, CON_2H_4.

841. **Urea** resembles the alkaloids by combining directly with acids to produce salts. It is a colourless crystalline substance, and is very soluble in water. It melts and begins to decompose at 132° C., evolving NH_3. Its specific gravity is 1·323.

842. **Strong HNO_3**, if it is added to solution of urea causes the separation of the crystalline nitrate: under the microscope these crystals are seen to be delicate rhomboidal scales

Strong solution of $H_2C_2O_4$ also causes a precipitate, which consists of needle-shaped crystals of oxalate of urea.

843. **KHO**, when it is heated with urea solution, slowly evolves NH_3, and yields K_2CO_3; the carbonate is detected by effervescing on the addition of an acid.

844. **$Hg(NO_3)_2$** yields a white precipitate in urea solution.

845. **KBrO-solution** causes a brisk evolution of nitrogen gas from urea and from compounds containing urea. The KBrO-solution must be freshly prepared, by adding Br-water to KHO-solution until the liquid retains a yellow colour after it has been thoroughly mixed.

A similar reaction takes place when KBrO-solution acts upon a urate or upon an ammonium salt.

846. **Biuret-test.**—If solid urea is heated for some time just above its melting-point, biuret is formed and NH_3 is evolved. If the residue is then allowed to cool and is extracted with water, and the solution is mixed with several drops of $CuSO_4$-solution, and then with NaOH-solution added drop by drop, a *violet* coloration is produced.

ANILINE, OR AMIDOBENZENE, C_6H_7N.

847. Pure Aniline is a colourless liquid of peculiar smell; it usually becomes brown by exposure to the air, but this is due to the presence of an impurity; it boils at 184·5°, and has a specific gravity of 1·0242. It is slightly soluble in water and in alcohol, and is readily soluble in ether and in chloroform.

848. Salts of Aniline do not give the following reactions. But the base may be liberated from the salt by treating the aqueous solution with KHO. The aniline will separate in oily drops, and these may be dissolved by shaking the liquid with ether. The evaporation of the ethereal solution leaves the aniline in the free state, in a condition suited for the following tests.

849. Bleaching-powder, if it is added in small quantity to a very dilute solution of aniline, produces a *mauve* coloration.

850. H_2SO_4 and $K_2Cr_2O_7$-solution, when they are added to aniline, produce a reddish coloration which changes to blue.

851. Chloroform and alcoholic solution of KHO, when they are warmed with aniline, evolve the intensely offensive smell of phenyl isocyanide.

X.—SUBSTANCES RELATED TO THE FOREGOING.

ETHER, OR ETHYL ETHER, $C_4H_{10}O$.

852. Pure ether is a very mobile, volatile, fragrant liquid, which burns with a bright flame. It has a boiling-point of 34·6° C., and a specific gravity of 0·7201. Ether dissolves in about ten times its own volume of water, and is miscible with alcohol and with other organic liquids.

ACETIC ETHER, OR ETHYL ACETATE, $C_2H_5(C_2H_3O_2)$.

853. **Pure Acetic ether** is a very mobile, fragrant liquid. It has a boiling-point of 77·5° C., and a specific gravity of 0·9072. It is somewhat soluble in water, and is readily soluble in alcohol.

854. **KOH.**—Acetic ether is hydrolysed by KOH in a manner which is typical of similar organic bodies:—

$$C_2H_5(C_2H_3O_2) + KOH = C_2H_6O + KC_2H_3O_2.$$

This change may be effected by simply warming the liquid for some time with KOH. When the resulting liquid is distilled, alcohol may be detected in the distillate (801), and acetate may be found in the distillation flask (670, *et seq.*).

CARBON DISULPHIDE, CS_2.

855. **Pure Carbon disulphide** is a mobile, highly refractive liquid with pleasant ethereal smell, but it usually emits an extremely repulsive odour which is due to the presence of impurity. CS_2 boils at 46° C., and has a specific gravity of 1 292. It is not miscible with water, and readily dissolves fatty bodies and oils. CS_2 burns with a blue flame, producing SO_2 (519) and CO_2 (505).

856. **Formation of Potassium xanthate.**—If CS_2 is heated with solution of KOH in alcohol, potassium xanthate ($C_2H_5KCOS_2$) is formed: the addition of $CuSO_4$-solution to this liquid produces a *yellow precipitate* of copper xanthate.

NITROBENZENE, $C_6H_5(NO_2)$.

857. **Nitrobenzene** is a pale yellow liquid smelling of bitter almonds: its specific gravity is 1·2, and it boils at 205°: it is insoluble in water, but is miscible with other organic liquids. See reaction in paragraph 790.

XI.—CARBOHYDRATES.

GRAPE SUGAR, DEXTRO-GLUCOSE, OR DEXTROSE, $C_6H_{12}O_6$.

859. **Dextrose** is a colourless soluble solid, with a sweet taste. It crystallises from its solution in alcohol in the anhydrous state, and these crystals fuse at 146° : from its solution in water it crystallises in a hydrated form, $C_6H_{12}O_6.H_2O$, which melts at 86°. Dextrose chars readily when it is heated; its specific gravity is 1·538, and its specific rotatory power is $[\alpha]_D + 52\cdot8$.

860. **Strong H_2SO_4.**—Dextrose is distinguished from other sugars by yielding only a *yellow solution* when strong H_2SO_4 is added to its strong solution, if the mixture is kept cool: other sugars become blackened by charring under these conditions.

861. **KHO.**—When dextrose is warmed with strong KHO-solution the liquid becomes *brown;* other sugars remain uncoloured by this treatment.

862. **Reduction of CuO.**—If a few drops of $CuSO_4$-solution are mixed with dextrose-solution and excess of KHO is then added, no precipitate is formed; but when the liquid is heated yellow cuprous hydrate is formed, and this is converted into red Cu_2O when the liquid is more strongly heated.

863. **Fehling's-solution** (864) may be added with advantage in applying the preceding test, instead of adding $CuSO_4$ and KHO separately.

864. Fehling's-solution is prepared as follows. Thirty-five grammes of crystallised $CuSO_4.5H_2O$ are dissolved in one litre of water, and the clear solution is kept in a stoppered bottle. 173 grammes of Rochelle-salt (KNa$\bar{T}$), and sixty grammes of NaHO, are dissolved in a litre of water; this solution is kept in a corked bottle. Equal volumes of these two solutions are mixed together in order to prepare the Fehling's-solution

865. Fermentation.—If clean freshly-washed *yeast* is added to dextrose-solution in the flask shown in fig. 73 (p. 202), and the temperature of the liquid is maintained at about 25° for an hour or more, fermentation will take place with the production of ethyl alcohol and carbon dioxide gas.

The escaping gas may be shown to be CO_2 by letting it bubble through lime-water (**505**, 4), and ethyl alcohol may be found in the liquid which remains in the flask by the reactions in paragraphs **801**, **802**.

This reaction by fermentation is given by other sugars.

866. Silver mirror.—If dextrose is gently heated with ammoniacal $AgNO_3$-solution (**694**, *Note*) in a glass vessel, a silver mirror will be produced on the interior of the glass.

867. Other Reduction Tests.—Dextrose differs from maltose and lactose by forming red Cu_2O when it is boiled with $Cu\bar{A}_2$-solution containing $H\bar{A}$. When dextrose is heated with solutions of KOH and of basic bismuth nitrate it yields a black precipitate.

868. When dextrose is boiled with KOH-solution and picric acid a *red* colour is produced.

Levulose, or Fructose, $C_6H_{12}O_6$.

869. Levulose is usually a colourless, difficultly crystallisable syrup. It is sweeter than dextrose, and is also more soluble in water and in alcohol. Its specific rotatory power is $[\alpha]_D - 102° - 0{\cdot}56.t°)$. It is fermented by yeast, but differs from dextrose by forming an insoluble compound with lime.

Cane sugar, or Sucrose, $C_{12}H_{22}O_{11}$.

870. Cane-sugar is a colourless crystalline substance, which is easily soluble in water and is intensely sweet in taste. It is only sparingly soluble in alcohol. Cane-sugar fuses at

160°, and is readily charred by heat. Its specific gravity is 1·593, and its specific rotatory power is $[a]_D + 66·5$. It is not readily fermented by yeast.

871 **Strong H_2SO_4** produces a deep brown coloration, which becomes ultimately black owing to the separation of carbon. The presence of much water prevents this change from occurring at ordinary temperatures.

872. **KHO** produces no coloration (861), and no silver mirror is produced (866). (Differences from glucoses.)

873. **Conversion into Glucose.**—Sucrose solution, when it is heated for a minute with a few drops of strong HCl, yields a mixture of equal quantities of dextrose and levulose: these glucoses may be detected by Fehling's-solution (864), after the acid liquid has been neutralised by KHO.

874. **Citric acid** in 5 per cent. solution, when boiled with sucrose, hydrolyses it forming glucose. (Distinction from maltose and lactose.)

MALTOSE, OR MALT SUGAR, $C_{12}H_{22}O_{11}.H_2O$.

875. **Maltose** is a soluble sweet sugar, resembling the glucoses by readily reducing Fehling's-solution (864), but differing from them by not reducing the acetic acid solution of $Cu\bar{A}_2$. It is readily fermented by yeast. Its specific rotatory power is $[a]_D + 138$. It differs from cane-sugar by being converted entirely into dextrose when it is heated with an acid, and by the reaction in paragraph 874.

LACTOSE, OR MILK-SUGAR, $C_{12}H_{22}O_{11}.H_2O$.

876. **Lactose** is much less soluble and less sweet in taste than the sugars which have been already described. It reduces Fehling's-solution (864), but does not reduce the acetic acid solution of $Cu\bar{A}_2$. It is not fermented by pure yeast. Its specific rotatory power is $[a]_D + 52·5$.

877. When lactose is boiled with $Pb\bar{A}_2$-solution and AmOH is added, a precipitate is formed which is first yellow and then red in colour.

CELLULOSE $(C_6H_{10}O_5)_n$.—Use Cotton-wool.

878 **Cellulose** is a colourless, insoluble, tasteless substance: it is unaffected by water and by most other solvents, even when it is boiled with them.

879. **Iodine reaction.**—Cellulose gives no reaction with iodine. After it has been treated with one of the following mixtures (*a*, *b*), however, iodine produces a blue colour with the product.

(*a*) A mixture of 90 parts of $ZnCl_2$-solution of 2·0 specific gravity, with 6 parts of KI and 10 parts of water.

(*b*) A mixture of 3 parts of strong H_2SO_4 with 2 parts of glycerol and 1 part of water. This mixture is rubbed with the cellulose in order to effect the necessary change.

880. **Cuprammonium hydrate**, made by dissolving freshly-precipitated $Cu(OH)_2$ in *strong* ammonia solution, dissolves cellulose: the cellulose is reprecipitated when an acid is added in excess to the solution.

STARCH.—Use Potato or Rice starch in powder $(C_6H_{10}O_5)_n$.

881. **Starch** is insoluble in cold water, but it yields a transparent liquid when it is *boiled* with water; this liquid becomes gelatinous as it cools, if much starch is present.

882. **Starch granules** may be identified by their appearance, when they are examined by a quarter-inch object-glass under the microscope: when they are viewed as transparent objects they usually appear round or oval, but they are occasionally irregular in shape. They usually show dark rings which are more or less concentric. Many kinds of starch-granules show a cross when they are examined by polarised light. These appearances are not visible in starch which has been exposed to heat.

883. **Iodine reaction.**—If starch is boiled with water, and a drop of iodine-solution is added to the *cold* liquid, it will assume an intensely *bluish-black* colour.

This colour disappears when the liquid is heated, but it frequently reappears when the liquid is cooled.

The colour is destroyed by the addition in excess of a reducing agent, such as H_2SO_3, $Na_2S_2O_3$, or H_2S · it is therefore prevented from appearing if any one of these substances is present in the starch-solution, until the iodine has been added in excess.

884. **Conversion into Glucose.**—When it is heated with dilute H_2SO_4 for some time, starch is converted into a mixture of dextrin and glucose, and finally into glucose alone. The glucose may be detected by means of Fehling's-solution, after the liquid has been neutralised (863).

DEXTRIN $(C_6H_{10}O_5)_n$.

885. **Dextrin** is a white powder, which is soluble in water, but is insoluble in *strong* alcohol. It is therefore precipitated from its aqueous solution by the addition of much alcohol. Its specific rotatory power is $[\alpha]_D + 200{\cdot}4$.

886. **Fehling's-solution.** — Dextrin is converted into glucose by being heated for some time with diluted H_2SO_4; when this solution is neutralised and heated with Fehling's-solution, red Cu_2O is precipitated (863).

887. **Iodine-solution** produces a red colour in the solution of most forms of dextrin.

XII.—ALBUMINOUS SUBSTANCES.

ALBUMEN.—Use White of egg.

Solution of Albumen is readily obtained by stirring or shaking the fresh unboiled white of egg with water, and then straining the solution through linen or allowing it to settle.

888. **Ignition with Soda-lime.**—Albumen evolves NH_3 when it is strongly heated with soda-lime; the NH_3 may be detected by the methods described in paragraph 145.

889. **Albumen is readily Coagulated** and precipitated as white flocks from its solution in water, either by boiling the solution, or by adding it to strong HNO_3 or to solution of $HgCl_2$. Albumen is less completely precipitated from its solution by solutions of $CuSO_4$, of alum and of certain other salts, than by solution of $HgCl_2$.

890. **Millon's test.**—**Solution of Mercurous nitrate,** prepared by dissolving 2 parts of mercury in 4 parts of HNO_3 (sp. gr. 1·4), imparts a bright red colour to albumen.

GELATIN.

891. **Gelatin** is readily soluble in hot water, and a hot strong solution "sets" on cooling. When solid gelatin is heated, it chars and produces an unpleasant smell. When it is heated with soda-lime it evolves NH_3.

892. **Tannic acid** solution precipitates gelatin from its solution (714).

Note.—Provision is made in the ordinary Analytical Tables in Sections V. and VI. for the detection of some of the more common Organic Acid-radicles. A further special Scheme for the Detection of Organic substances will be found in paragraphs 1093–1106. Many organic substances, however, must be tested for by special reactions (647–892).

SECTION V.

ANALYSIS OF A SIMPLE SUBSTANCE CONTAINING ONE METAL AND ONE ACID-RADICLE.

This Section may be passed over by a student who is learning the full analytical course, and the full course of analysis in Section VI. may be taken up at once.

Examples of Substances for Analysis by this Section are given in paragraph 1169

The Numbers in the Text which are Inclosed in Brackets refer to the paragraphs in which tests or processes to be employed by the student are fully described.

INTRODUCTORY REMARKS.

930. **Preliminary Examination.**—Before proceeding to detect the Metal or Acid-radicle in a Simple Substance by a Systematic Analysis of its solution, it is best to make a few Preliminary Experiments upon the substance in the solid state (936–941). These will generally give some idea as to what substance is present, and will occasionally detect with certainty the Metal or the Acid-radicle, or possibly both of them.

Systematic Examination for the Metal.—Even if the composition of the substance has been proved by this Preliminary Examination, it is usually best to proceed to the

Systematic Analysis in order to confirm the results obtained. This must be preceded by the process of dissolving the substance in water or in acid (935), if it is not already in the liquid form. To the solution thus obtained, the Group-Reagents are then added in a suitable order. The Analytical Group to which the metal present belongs (931, 942) will thus be determined.

In the following Table (931) the metals are classified in their Analytical Groups. Each Group is headed not only by its distinctive number and by its distinctive name, but also by the name of the Group-Reagent which serves to precipitate its members and to separate them from those of the succeeding Groups.

When the Group to which the Metal present belongs has been ascertained, the metal is identified by the properties or appearance of the Group-precipitate. Its presence is then confirmed, if necessary, by special tests which are applied either to the precipitate itself or to another portion of the *original solution* * (947–951).

If the Acid-radicle has not been detected during the examination for the Metal, it must be specially tested for. With this object a few preliminary experiments are first performed (938–941). If these do not serve to detect the Acid-radicle, it must be looked for by the application of special tests (952–961).

Entry of Results.—While the analysis is progressing, each result must be fully entered in the note-book *as soon as the experiment has been performed;* the form of entry is the same as that which is adopted for the directions in the text and for the Analytical Tables.

In these Simple Analyses, One Metal and One Acid-radicle only are to be looked for. The examination for the

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* By the *original solution* is meant the solution of the substance, or the substance itself if a liquid is under analysis, to which no reagent has been added.

Metal is therefore carried no further, when one metal has been detected with certainty; and the examination for the Acid-radicle is similarly at an end when the presence of one such radicle has been satisfactorily proved.

Two Metals may occasionally be present, as when an alum [*e.g.*, $KAl(SO_4)_2$] or tartar-emetic [$K(SbO)\bar{T}$] is given for analysis.

Two Acid-radicles may also occasionally be present in the liquids which are given for analysis, since a salt which is insoluble in water may be dissolved in an acid.

But the student should always be informed of the presence of two Metals or Acid-radicles, if it in any way complicates the analysis. This will not usually be the case, since in the most commonly occurring instance, that of a phosphate dissolved in HCl, the PO_4 is detected in Group III. and accordingly no other Acid radicle is looked for.

It must also be remembered that the substances which are given for analysis are liable to contain impurities; these impurities will, however, usually yield only slight indications, which will not mislead the analyst. If in the course of analysis *only a slight* result is obtained, it is well to proceed until some substance is detected in larger quantity: against the slight result, *Trace of* ——, is then entered.

The separation and washing of a precipitate by *decantation* (94, 97) is recommended whenever it is possible.

931. ANALYTICAL CLASSIFICATION OF THE METALS.

GROUP I.	GROUP II.	
Metals whose chlorides are insoluble in water and in dilute acids, and which are therefore precipitated from their solutions by hydrochloric acid.	Metals whose sulphides are insoluble in dilute acids, and which are therefore precipitated from *acid* solutions by hydrogen sulphide	
Hydrochloric acid, or Silver Group.	**Hydrogen sulphide Group.**	
	GROUP II A **Copper Group.** Sulphides insoluble in NaHO and in Am_2S	GROUP II B. **Arsenic Group.** Sulphides soluble in NaHO and in Am_2S
[Lead . Pb] Silver Ag Mercurosum Hg'	Mercuricum . Hg'' [Lead . Pb] Bismuth Bi Copper . Cu Cadmium Cd	Tin . . Sn Arsenic As Antimony . . Sb

GROUP III.		GROUP IV.	GROUP V.
Metals which are precipitated by NH_4OH as hydrates from their solutions containing ammonium chloride, or as sulphides or hydrates when ammonium sulphide or hydrogen sulphide is subsequently added.		Metals whose carbonates are precipitated by addition of ammonium carbonate in the presence of ammonium chloride.	Metals whose solutions are not precipitated by any of the foregoing Group-reagents in the presence of ammonium chloride.
Ammonium hydrate and Ammonium sulphide Group.		**Ammonium carbonate, or Barium Group.**	**Potassium Group.**
GROUP III.A. **Ammonium hydrate, or Iron Group.**	GROUP III B. **Ammonium sulphide, or Zinc Group.**		
Aluminium . Al Iron . . Fe Chromium . Cr	Zinc . . Zn Manganese . Mn Nickel . . Ni Cobalt Co	Barium . . Ba Strontium . Sr Calcium . Ca	Ammonium NH_4 Potassium . K Sodium . Na Magnesium Mg

DIRECTIONS FOR THE ANALYSIS OF A SIMPLE SUBSTANCE.

The substance given for analysis may be either a solid or a liquid. If it is a solid examine it by paragraphs 932, *et seq.*, or by paragraph 969 if it is metallic in appearance, omitting paragraph 937. If a liquid is to be analysed begin at paragraph 937.

THE SUBSTANCE IS A SOLID.

The Appearance and General Properties of the substance must be carefully observed and noted down.

932. **Note its Colour.** The following are some of the most commonly occurring coloured substances.—

Blue · hydrated cupric salts, and anhydrous cobalt salts.
Yellow. normal chromates, precipitated HgO, CdS, As_2S_3, SnS_2.
Brownish-yellow ferric salts, PbO
Orange-red. certain dichromates, Sb_2S_3.
Red: HgO, HgI_2, HgS, Pb_3O_4.
Green. Cr_2O_3, $CuCl_2$, salts of Ni and Cr. *Pale green:* ferrous salt.
Reddish-pink: hydrated cobalt salts. *Pale pink:* manganous salts.
Dark brown. PbO_2, Fe_2O_3. *Light brown:* PbO, Bi_2O_3, CdO.
Black: Sb_2S_3, CuO, MnO_2, FeS, Co_3O_4
White: anhydrous salts of Cu and Fe″, and many other powdered substances.
Colourless. a large number of colourless crystalline bodies exist.

933. **Any characteristic Smell** of the substance is noted. Observe also whether it is in **powder, crystals,** or **non-crystalline pieces**, examining the substance with the assistance of a lens if necessary. Note also metallic appearance, hardness, or attraction by magnet.

934. **Powder the substance** as finely as possible (988), before subjecting it to systematic analysis. Then ascertain its solubility and examine the substance further, as is directed in paragraph 935.

DETERMINATION OF THE SOLUBILITY OF THE SUBSTANCE.

935. **Since the Method of Examination** of a solid substance depends upon whether it is soluble or insoluble in water or in acids, the following experiments are tried before the analysis is commenced.

Place a small quantity of the *finely-powdered* substance in a broad test-tube or in a boiling tube; then fill the tube about one-third with water, and heat the liquid to boiling. Refer to figure 38 (85) for other methods of dissolving a substance.

<table>
<tr>
<td rowspan="4">**1. The Substance dissolves.**
After examining a portion of the solid substance by the preliminary tests (936 and 938–941) proceed to examine the colour and reaction of the liquid to litmus by I, II (937), and refer to paragraphs 942 and 952 for directions for analysis.</td>
<td colspan="4">**The Substance does not dissolve:** heat a fresh portion with dilute HCl to boiling. If it does not dissolve, decant and boil the residue with strong HCl —</td>
</tr>
<tr>
<td rowspan="3">**2. The Substance dissolves.**
Absence of Group I, except possibly Pb.
After examining a portion of the solid substance by the preliminary tests (936 and 938–941) note the colour of the HCl solution (937, I), and proceed to examine it for the metal by paragraph 942, and for the acid-radicle by pars. 952 *et seq.*</td>
<td colspan="3">**The Substance does not dissolve:** put aside the test-tube containing the substance with HCl, and stir a fresh portion of the substance with a little dilute HNO_3; if it is not dissolved, boil. If dilute HNO_3 does not dissolve it, heat with a little strong HNO_3 —</td>
</tr>
<tr>
<td rowspan="2">**3. The Substance dissolves.**
After examining a portion of the solid substance by the preliminary tests (936 and 938–941) note the colour of the solution in HNO_3 (937, I) and examine it for the metal by paragraph 942, and for the acid-radicle by par. 952.
Note. — Before testing for Group II., add strong HCl and boil down considerably, in order to remove the excess of HNO_3.</td>
<td colspan="2">**The Substance does not dissolve:** mix together the contents of the two tubes in which the substance was heated with HCl and with HNO_3, and heat the mixture.—</td>
</tr>
<tr>
<td>**4. The Substance dissolves**
After examining a portion of the solid substance by the preliminary tests (936 and 938 - 941) examine the solution in the mixture of HNO_3 and HCl for the metal by paragraph 942. See *Note*, column 3.
Examine for the acid-radicle by par. 952.</td>
<td>**5. The Substance does not dissolve.**
Examine a fresh portion of the substance by paragraphs 970–974.</td>
</tr>
</table>

PRELIMINARY EXAMINATION OF A SIMPLE SOLUBLE SOLID FOR THE METAL.

936. **The Following Table** is thus arranged. In the first column is placed the experiment to be made; in the second a list of the results, any one or more of which may be observed; and in the third the inferences to be drawn from these results.

Confirmatory or supplementary experiments are occasionally entered; they are, however, only to be tried, when the result which immediately precedes them in the second column has been noticed.

A more complete Preliminary Examination will be found in paragraphs 989-995.

Experiment.	Observation	Inference.
Exp I.—Heat a portion of the substance in an ignition-tube, or in a small dry test-tube, first gently and at last to redness.	**I. The substance does not change.**	Absence of volatile, fusible and organic substances, and of water.
	II The substance changes:—	
	1 Drops of water condense in the top of the tube	Presence of H_2O.
	2 The substance fuses easily .	Probable presence of a salt of Na or K, or of Ba, Sr, Ca, Mg.
	3 The substance sublimes .	Presence of a salt of NH_4, As, or Hg. possibly also of $H_2C_2O_4$, of S (yellow), and of I (purple vapour).
Confirmatory.—Boil another portion of the substance with KHO solution, added in excess.	A smell of NH_3 is evolved, and white fumes appear when a rod moistened with strong HCl is held to the mouth of the tube.	Presence of NH_4. Confirm by NaHT (147) and proceed to paragraph 938.
Confirmatory.—Mix another portion of the substance with about three times as much dry powdered Na_2CO_3 and KCy, and heat the mixture in an ignition tube.	A grey sublimate of minute metallic globules, best seen by a lens.	Presence of Hg. Pour AmHO upon the original substance, if it blackens Hg' is present as a mercurous compound.
	A black mirror-like sublimate	Presence of As.
	4. The substance blackens, and gives off CO_2, which makes a drop of lime-water on a glass rod milky.	Presence of an organic substance.
	The cool residue in the tube effervesces with HCl, whereas the original substance does not.	**An organic salt of K, Na, Ba, Sr, or Ca.**

Experiment	Observation.	Inference.
	5. Gas or vapour is given off: smell the vapour, and test with spark and flame:—	
	Red nitrous fumes, recognised by their smell and colour.	From nitrate of a heavy metal.
	SO_2, known by its smell and by turning $K_2Cr_2O_7$ solution green.	From combustion of free S or a sulphide, possibly also from a sulphate, sulphite, or thiosulphate
	Cl, Br, I, known by smell and colour: Br and I by action on starch paste, and Cl by bleaching moist litmus.	From certain chlorides, bromides, or iodides, also free I
	A slip of wood with a spark at its end glows brightly or bursts into flame	O from chlorate or nitrate, or N_2O
	The flame has a greenish halo	N_2O from NH_4NO_3.
	The gas can be lighted and burns at the mouth of the tube, note the colour of the flame:—	
	Pale greenish-yellow: the gas burns explosively, and nitrous fumes are seen	Presence of NH_3 from strongly heated NH_4NO_3.
	Bright white, the unburnt gas smells of garlic.	Presence of PH_3, probably from a hypophosphite (624)
	Peach-blossom coloured flame, grey sublimate of Hg	Presence of Cy, from $HgCy_2$
	6. The substance changes colour:—	
	Yellow, hot. *White*, cold	ZnO
	Yellow-brown, ,, *Yellow*, ,,	PbO, SnO_2 or Bi_2O_3.
	Dark red or *brown*, ,, *Red brown*, ,,	Fe_2O_3
EXP II Dip into the powdered substance a moistened loop of platinum wire, and hold the loop in the Bunsen-flame; moisten with strong HCl, and again hold the loop in the flame	**I. The flame is not coloured,**	Probable absence of the substances enumerated below.
	II. The flame is coloured:—	
	Intense yellow,*	Presence of Na.
	Pale lilac,* appearing *crimson-red* through the indigo-prism	,, K.
	Red: Appearing *green* through the indigo-prism	,, Ca.
	Red: Appearing *intense red* through the indigo-prism	,, Sr
	Green: *Yellowish-green*	,, Ba.
	Green: *Bright green*, with *blue* centre after moistening with HCl	,, Cu.
	Green: *Bright green*	,, B_2O_3.
	Blue: *Pale blue, livid*	,, $HgCl_2$, $ZnCl_2$, $SnCl_2$, As, Sb, Pb
	Blue: *Vivid blue*	,, $CuCl_2$, $CuBr_2$.

*** If the coloration for Na or K is intense, no further examination for the metal is necessary, but the presence of K should be confirmed by stirring with $NaH\bar{T}$ (142). Proceed to 938. If the coloration is slight, a trace only of the metal is present, and the examination for another metal must be continued.**

Experiment.	Observation.		Inference.
Exp. III. Heat a portion of the substance in a cavity scooped on charcoal, in the blowpipe-flame.	1. The charcoal "deflagrates," or burns rapidly.		Presence of a nitrate or chlorate.
	2 A white mass is left on the charcoal, which does not fuse, but shines brightly when it is strongly heated		Presence of Ba, Sr, Ca, Mg, Al, or Zn
Confirmatory. — Detach a small portion of the white mass, place it on turmeric - paper and moisten it with a drop of water	The paper becomes brown in a short time where the substance rests.		Presence of Ba, Sr, Ca, or possibly of Mg.
Confirmatory —If the moist residue is not alkaline to test-paper, moisten the remainder on the charcoal with $Co(NO_3)_2$ solution and heat it again strongly in the blowpipe-flame	A *blue* residue		Presence of Al, or of a phosphate, arsenate, silicate, or borate
	A *pink* residue		Presence of Mg
	A *green* residue . . .		Presence of Zn, or possibly Sn
	3 The residue is not white and does not consist of globules of metal, neither is there any incrustation formed upon the charcoal, proceed to Exp IV		
	4 The residue contains metallic globules or an incrustation forms on the charcoal, proceed to Exp V, omitting Exp IV.		
Exp IV. Fuse a small quantity* of the substance into a clear colourless borax bead first in the inner then in the outer flame, noting in both cases the colour of the bead while it is hot and when it is cold.	**I. The bead is colourless after it has been heated in each of the flames**		Absence of the metals mentioned below.
	II. The bead is coloured.—		
	In outer flame	In inner flame	
	Green, hot, *blue*, cold	*Red* or *colourless*	Presence of Cu
	Blue, hot and cold	*Blue*, hot and cold	„ Co
	Brown or *dingy purple*, hot, *light brown*, cold	*Grey* or *black*, hot and cold	„ Ni
	Brown, hot, *yellow*, cold	*Bottle green*, hot and cold	„ Fe †
	Green,† hot and cold	*Green*, hot and cold	„ Cr
	Purple, hot and cold	*Colourless*, hot and cold . .	„ Mn
Confirmatory — The presence of Cr or Mn may be confirmed, by fusing a portion of the substance on platinum foil or wire with Na_2CO_3 and KNO_3	A *yellow* mass on cooling		„ Cr.
	A *bluish-green* mass on cooling .		„ Mn.

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* It is necessary to adjust carefully the quantity of substance which is fused into the bead, if too much is used, the bead will often appear opaque, if too little is employed, one of the metals may escape detection It is best to fuse only a minute quantity into the bead, and then to add more if no colour, or only a faint colour, is produced

† If Cr is present as a chromate, it gives a bead which is *brown* whilst hot, after it has been fused in the outer blowpipe-flame ; but this brown colour is not reproduced when the bead has been changed to green by being heated in the inner flame, and is then again heated in the outer flame. This distinguishes the brown chromate bead from the brown iron bead

Experiment.	Observation.		Inference.
Exp. V.—Mix the substance in a small cavity on wood-charcoal with Na_2CO_3 and KCy, and heat strongly in the inner blowpipe-flame for several minutes. If metallic globules are obtained, detach one and strike it sharply on the bottom of an inverted mortar with the pestle and note whether it is crushed to powder (*brittle*) or merely flattened out into a cake (*malleable*) If the globules are white and malleable, take one upon the point of a penknife and see if it will mark paper as a black-lead pencil does	Metallic scales or globules are obtained, or an incrustation forms on the charcoal.—		
	Globules	*Incrustation*	
	White and *brittle.*	*White* .	Presence of Sb.
	White and *brittle*	*Yellow* . .	" Bi
	Red and *malleable*	*None* . .	" Cu.
	White and *malleable* marking paper easily.	*Yellow* .	" Pb.
	White and *malleable* not marking paper and readily fusible	*None* .	" Sn.
	White and *malleable* not marking paper, fusible only with difficulty	*None* . .	" Ag.
	None	*Yellow* whilst hot, *white* when cold, becomes green when moistened with $Co(NO_3)_2$ solution and reheated in the outer flame,	" Zn.
	None . .	*Brown* . .	" Cd
	None	*White;* on smelling the charcoal a smell of onions is perceived.	" As.
Exp. VI.—Fuse the substance in a colorless bead of $NaAmHPO_4$.	Particles float about undissolved in the bead		Presence of silica or of silicate.

After the above Preliminary Examination for the Metal has been completed, the Preliminary Examination for the Acid-radicle (938, *et seq.*) is made. The solution of the substance is then prepared according to the directions in paragraph 935, and is subjected to the full systematic Examination (942, *et seq.*).

937. Preliminary Examination of a Liquid.

Experiment.	Observation.	Inference.
Exp. I.—Note the Colour of the liquid. *Note*.—If the colour has to be observed by night, it should be examined by the white light obtained by burning a piece of magnesium ribbon. Delicate tints are best seen by looking through some thickness of the liquid at a sheet of perfectly white paper.	**I. The liquid is Coloured:—**	
	Pink { Very delicate . . .	Presence of Mn or dilute Co solution.
	Pink { Intense . . .	Presence of Co
	Green { Very delicate	„ Fe$''$, Ni, or Cr in dilute solution
	Green { Intense . . .	„ Ni, Cr, or $CuCl_2$
	Yellow { Reddish-yellow . .	„ Fe$'''$ or a bichromate
	Yellow { Light yellow . .	„ neutral chromate.
	Blue . . .	„ Cu
	Violet or *purple* . .	„ Cr, or a permanganate.
	II. The liquid is Colourless . .	Absence of Co, Ni, Cr, Cu, &c
Exp. II. — Dip pieces of turmeric-paper and of blue litmus-paper into the liquid.	**It is Neutral** . . . . . . (Not affecting the colour of either paper)	Absence of free acids and alkalis, and of all salts but some of those of Am, Na, K, Mg, Ba, Sr, Ca, Ag.
	It is Acid . . (Turning blue paper red) If no effervescence occurs when Na_2CO_3 is added to a part of the solution and the liquid is warmed, free acid and acid salt are absent.	Presence of a free acid, of an acid salt, or of a salt with acid reaction
	It is Alkaline . . . (Turning turmeric brown) If the colour of the paper becomes only slightly changed, the probable presence of an alkaline salt may be inferred.	Presence of a hydrate of K, Na, Am, Ca, Sr, or Ba, or of a salt with alkaline reaction.

Experiment	Observation.	Inference.
Exp. III. Evaporate (87) some of the liquid upon a thin watch-glass,*heated very gently by supporting it some inches above a small flame upon a piece of wire gauze, or by means of a sand- or water-bath. If a residue remains on the glass bring this gradually into the flame and heat it more strongly	**A Residue is left** . . .	Presence of some dissolved solid.
	When it is strongly heated, the residue blackens and emits a smell of burning	Presence of organic substance.
	The smell resembles that of charred sugar .	Presence of $\overline{\text{T}}$, probably.
	Note.—If the cold residue, after ignition, effervesces with a drop of HCl, whereas the unignited residue did not, the organic acid-radicle is united with Ba, Sr, Ca, Mg, Na, or K.	
	The results in Exp I, par 936, may be here observed and should be noted.	
	Keep this residue for Exp. VI	
	No Residue is left . . . If the liquid is also colourless, tasteless, and without action on litmus-paper, it must consist only of distilled water, and no further examination of it need be made If the liquid is alkaline, NH_3 must be looked for, if it is acid, H_2CO_3, H_2SO_3, HCl, HNO_3, H_2SO_4, and other volatile acids only need be tested for.	Absence of any dissolved solid.
Exp. IV. Add excess of strong NaHO or KHO solution and boil (145).	A smell of NH_3, moist turmeric is turned brown, and a rod moistened with strong HCl produces white fumes when it is held in the mouth of the test-tube	Presence of NH_4 No further test need be tried for a metal. Proceed to par 938.
Exp. V. The flame coloration (936, II.) is then tried by dipping the platinum wire into the liquid, which has been first concentrated by evaporation, if it is very dilute		
Exp. VI. Some of the residue from Exp. III. (above) is then fused into a borax bead (936, IV.).		

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* A piece of thin glass from the side of a broken flask is a cheap and useful substitute for a watch glass.

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PRELIMINARY EXAMINA-

938. **Pour dilute H_2SO_4 in excess** upon the substance in a test-tube,
A gas is evolved, as is shown by the occurrence of effervescence

1. Colourless gas without Smell.	**2. Pungent gas smelling of Burning sulphur.**	**3. Gas with a most Fetid smell.**	**4. Yellow gas smelling of Chlorine.**
A drop of lime- or baryta-water, held in the tube upon the end of a glass rod, becomes milky :—	A drop of $K_2Cr_2O_7$ solution, held in the tube upon the end of a glass rod, becomes green —	A slip of filter-paper moistened with alkaline solution of $Pb\bar{A}_2$, and held in the tube, is blackened .—	A slip of moist litmus-paper held in the tube is bleached —
Presence of Carbonate.	**Presence of Sulphite.**	**Presence of Sulphide.**	**Presence of Hypochlorite.**
	2*a* The SO_2 gas is given off only after a time or when the liquid is warmed, and yellow S separates .— **Presence of Thiosulphate.**		

939. **Add a little strong H_2SO_4** to a fresh portion of the substance,
results enumerated below occurs *†: if none of them is noticed, pro-

1. Fumes are evolved with Pungent smell and Acid reaction to litmus-paper: the Liquid is not coloured :— **Presence of Chloride or Nitrate.**		**2. The liquid becomes Yellow or Red, and a Chlorous smell is given off. when the tube is warmed a crackling noise or slight explosion is produced .—** **Presence of Chlorate.** *Confirmatory.*—To a portion of the cold solution of the substance in water add a few drops of dilute indigo solution, then pass some SO_2 the blue colour of the indigo is destroyed if **Chlorate is present.**
Confirmatory. — Hold in the fumes a glass rod moistened with a solution of $AgNO_3$ acidified with HNO_3 the solution on the rod becomes milky — **Presence of Chloride.** Confirm by par 955.	*Confirmatory* — Drop into the acid some small pieces of Cu, reddish-brown gas is evolved .— **Presence of Nitrate.** Confirm by par 537.	

940. **Fluoride** —After the tube which contains the strong H_2SO_4 and
the liquid into a porcelain dish for the test in paragraph 941. Rinse
and dimmed :—**Presence of Fluoride** is shown. If the presence of

941. **Borate.**—Add alcohol to the liquid in the porcelain dish from
alcohol; if the flame has a greenish tinge it shows the **Presence of**

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* If Pb, Ba, Sr or Ca is present, the addition of H_2SO_4 will cause a precipitate
† It is best to compare this smell with that which is evolved when $Na\bar{A}$
somewhat fragrant smell of ether when it is heated, and this might be mistaken

TION FOR THE ACID-RADICLE.

and observe the effect which is produced; * then heat to boiling: or by the evolution of a characteristic smell; see below.—

5 **Reddish-brown fumes are evolved,** which are most easily seen by looking down the tube at white paper. When a little *cold dilute* H_2SO_4 and $FeSO_4$ solution are added to a fresh portion of the original substance, a deep brown liquid is produced — **Presence of Nitrite**	6 **Smell of Almonds.** Test for a cyanide and a ferro-, ferri-, and sulphocyanide in the original substance by par 955 7 **Smell of Vinegar** Refer to par 939 (4) below	**No Gas is evolved.—** Absence of carbonate, sulphite, thiosulphate, sulphide, hypochlorite, nitrite. Pass on to par 939

mix well by shaking, then heat gently and note whether one of the ceed to paragraphs 940 and 941.

3. **A Reddish-brown or Violet colour** is produced in the acid, and no explosion or crackling occurs when it is warmed — **Presence of Bromide or Iodide.** If coloured vapour is evolved when the mixture is heated (see Note), hold in the vapour the end of a glass rod covered with moist starch powder. the starch becomes brown.— **Presence of Bromide.** The starch becomes bluish-black — **Presence of Iodide.** *Note.*—The vapour is much more easily obtained if the substance is mixed with MnO_2 powder, before H_2SO_4 is added and the mixture is heated	4 **A smell of Vinegar is** noticed.— **Presence of Acetate.** *Confirmatory* — Add to a fresh portion of the substance some alcohol, then strong H_2SO_4; a fragrant smell proves the **Presence of Acetate.**†	5 **A mixture of** CO and CO_2 **gases is given off.** found by a drop of lime-water held in the tube being turned milky, and by the CO burning with a blue flame when it is kindled.— **Presence of Oxalate**	6 **The substance Blackens and evolves gases, amongst** which SO_2 is **recognised by its smell and by its action on** $K_2Cr_2O_7$ solution.— **Probable Presence of Tartrate.** Confirm by par. 958.

the substance has been heated, let it stand for some time; then pour out the tube, and dry the inside thoroughly; if it is seen to be corroded fluoride is decisively indicated here, it will require no confirmation. par. 940, mix the contents by stirring, heat the dish and kindle the **Borate.** Confirm by par. 957.

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of the insoluble sulphate; this may be disregarded.
is similarly treated, since a mixture containing alcohol and H_2SO_4 evolves a for the more fragrant smell of ethyl acetate.

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GENERAL TABLE FOR THE

942. If the metal has not been detected in the Preliminary Group it belongs [see Table, par. 931], and then trying special

<table>
<tr><td colspan="3">A part of the solution of the substance (935), <i>if it is acid</i> (943), is mixed with dilute HCl</td></tr>
<tr><td rowspan="3">The precipitate may be—
AgCl—white.
Hg_2Cl_2— „
$PbCl_2$- „
Examine for the metal by Table I (947)</td><td colspan="2">Into the same part of the solution H_2S is passed (945), or H_2S the liquid is heated to boiling –</td></tr>
<tr><td rowspan="2">The precipitate may be—
HgS—black
PbS— „
Bi_2S_3— „
CuS— „
CdS—yellow
SnS—brown
SnS_2—yellow
Sb_2S_3—orange
As_2S_3—yellow
Examine for the metal by Table II (948)</td><td>To a Fresh portion of the Solu-
If a precipitate is pro- ride (940), or a borate to dryness, ignite the and add AmCl and ex-
If the removal of the acid- metal by this Table, or
If AmHO added after AmCl the original solution are presence of <i>phosphate</i>,</td></tr>
<tr><td>The precipitate may be—
$Al_2(HO)_6$—{ Colourless and transparent
$Cr_2(HO)_6$—pale green
$Fe_2(HO)_6$—reddish-brown
$Fe(HO)_2$—{ dingy green, turning brown in the air
Examine for the metal by Table III A (949)
If the precipitate is opaque or white and has none of the above appearances, refer to par 962</td></tr>
</table>

If no metal can be found, the sub-

NOTES TO THE GENERAL TABLE.

943. **If the liquid is Neutral or Alkaline,** a portion of it is made just acid by slowly dropping in dilute HNO_3 (628, 629).

If this produces no precipitate, or a precipitate which dissolves when more HNO is added, another portion of the liquid is examined by the above Table.

If a precipitate forms and is not dissolved when more HNO_3 is added, it may consist either of colourless gelatinous H_4SiO_4 from a silicate; or of finely-divided S, which is precipitated *white* from a polysulphide, and *yellow* from a thiosulphate; or of SnO_2, or metastannic acid, or Sb_2O_5 (all white). It may also possibly be SnS_2, As_2S_3 (both yellow); or Sb_2S_3 (orange-red), these sulphides are precipitated from a sulphostannate, a sulpharsenate, and a sulphantimonite respectively.

If H_4SiO_4 separates, it is best to evaporate the acidified solution quite to dryness and then heat the residue with dilute HCl. If an

EXAMINATION FOR THE METAL.

Examination, it is most rapidly found by ascertaining to which tests for each member of the Group.

(944), if any precipitate forms, more HCl is added and the liquid is heated —

water is added to it (946) the liquid is then diluted, and, if no precipitate has formed,

tion AmCl is added, then AmHO is added in excess.—

duced and either an organic substance (936, I., 4, 939, 5, 6), a silicate (936, VI.), a fluo-
(832) has been found, acidify another part of the original solution with HCl, evaporate
residue, and dissolve it when cool in dilute HCl, filter, if necessary, from SiO_2 (599),
cess of AmHO

radicle by the above treatment does not lead to the satisfactory detection of the
if chromate is present (946), it may be necessary to proceed as is directed in par 962
has produced a precipitate and the above acid-radicles are not present, a few drops of
added to some $AmHMoO_4$ and warmed if a yellow precipitate forms, indicating the
proceed to par 962

To the same part of the solution add a few drops of Am_2S, or H_2S water —

[*Note* —Before Am_2S is added the liquid may be *blue*, showing *presence of Ni*, or *violet* from dissolved $Cr_2H_6O_6$, the latter must be precipitated by boiling the liquid for a long time in a porcelain dish before Am_2S is added]

<table>
<tr><td rowspan="3">The precipitate may be—
ZnS--white
MnS- pink
NiS—black
CoS-- „
[FeS-- „]
Examine for the metal by Table III B (950)</td><td colspan="3">To the same portion of the solution add Am_2CO_3.--</td></tr>
<tr><td rowspan="2">The precipitate may be —
$BaCO_3$—white
$SrCO_3$— „
$CaCO_3$-- „
Examine for the metal by Table IV (951)</td><td colspan="2">To the same part of the solution add Na_2HPO_4, warm gently and shake well:--</td></tr>
<tr><td>A white crystalline precipitate proves --
Presence of Mg.</td><td>The metal still present may be NH_4, Na, K; it should have been detected already in the Preliminary Examination by Tests I. or II. (par 936).</td></tr>
</table>

stance is probably an acid (937, II.).

insoluble residue remains, this confirms the presence of a silicate. The residue is filtered off and the filtrate is tested for the metal by the above General Table.

Any other substance which remains undissolved by the dilute HNO_3 is filtered off. It may generally be recognised by its appearance; SnO_2 becomes *yellow*, and Sb_2O_5 *orange*, when it is moistened with H_2S-water metastannic acid becomes golden yellow when it is moistened with $SnCl_2$-solution.

944. **Any Change which occurs on the Addition of HCl** should be carefully noted. Gas may be evolved and recognised by the statement given in paragraph 938; a *yellow* solution may become *reddish-yellow*, and this change renders probable the presence of a neutral chromate.

945. **Before H_2S is added, the liquid must be heated**; and if a smell of Cl or of SO_2 is evolved, or a reddish-brown gas is perceived, the solution is boiled down nearly to dryness, then dilute HCl is added and H_2S is passed into the acid solution. Gelatinous H_4SiO_4 may separate during the process of evaporation (943).

946. The Following Changes, which occur on the gradual addition of H_2S, are noteworthy. If the precipitate is *white* at first, then *brown*, and at last *black*, the presence of Hg'' is shown. If the solution is strongly acid and gives a *red* precipitate, which becomes *black* when the liquid is diluted with H_2S-water, Pb is present.

A separation of *white* sulphur may occur. if it is accompanied by one of the following changes of colour in the solution, the change should be noted down, since it is characteristic :—

A *reddish-yellow*	solution	changing to	*pale green*	indicates	a ferric salt
A *reddish-yellow*	,,	,,	*bright green*	,,	chromate
A *green*	,,	,,	*colourless*	,,	manganate
A *purple*	,,	,,	*colourless*	,,	permanganate
A *green alkaline*	solution	*becoming purple when diluted or acidified, and colourless with* H_2S		,,	manganate

The precipitate of S is known by being perfectly white and by running through a filter; it need not be further examined.

If a yellow precipitate forms only when the liquid is boiled, it may consist of either As_2S_3 from an arsenate, or of SnS_2 from a stannic salt.

TABLE I.—SILVER GROUP.

947. *A white precipitate* forms when HCl is added. It may consist of AgCl, $PbCl_2$, or Hg_2Cl_2. The precipitate is either not dissolved when it is boiled with HCl: or it dissolves and reappears in crystals when the liquid is cooled, showing the *presence of Pb.*

Let the precipitate settle and decant the liquid: then boil the precipitate in the test-tube with AmHO :—

1. *The precipitate dissolves,* at least in part, see *Note* below.— *Presence of Ag.*	2. The precipitate does not dissolve:—	
	The colour of the precipitate is unchanged:— *Presence of Pb.*	*The precipitate becomes black.*— *Presence of Hg'* (*Mercurosum*).
Confirm by adding excess of HNO_3 to the AmHO solution, a milkiness appears. ——— *Note.*—Coagulated AgCl often dissolves only slowly in AmHO.	Confirm by decanting the liquid, dissolving the precipitate in $H\bar{A}$, and adding K_2CrO_4 to the solution; a yellow precipitate forms.	Confirm by immersing a bright slip of copper in a portion of the original solution, made acid with a few drops of HNO_3; a grey deposit forms on the Cu, which becomes white and bright when it is rubbed:—

948. Note the colour of the precipitate yielded by H_2S:—

<table>
<tr><td colspan="4">1 <i>The precipitate is dark brown or black</i>: it may consist of SnS, CuS, HgS, Bi_2S_3, PbS.
Add pure NaHO in excess to a small quantity of the liquid and precipitate, or of the precipitate alone after decanting the liquid if possible, and boil:—</td></tr>
<tr><td rowspan="2"><i>The precipitate dissolves</i>: to a portion of the NaHO solution add HCl in excess, brown SnS is reprecipitated. Boil another portion of the precipitate after adding a little yellow Am_2S, then add HCl in excess, a yellow precipitate of SnS_2 — <i>Presence of Sn''</i> (Stannosum).
The presence of Sn'' should be confirmed by adding $HgCl_2$ to a part of the original HCl solution, a white precipitate shows <i>Presence of Sn''</i>.</td><td colspan="3"><i>The precipitate does not dissolve</i>: it may consist of CuS, HgS, Bi_2S_3, PbS.
α <i>The original solution is blue or green</i>, and becomes intensely blue on addition of excess of AmHO.—<i>Presence of Cu</i>. Confirm by adding a few drops of H_2SO_4 to the original solution, and dipping into it a bright knife blade or piece of steel, a red film of Cu deposits.
β <i>The original solution is colourless</i>. Test in separate portions for Hg, Bi, Pb by the following special tests:—</td></tr>
<tr><td>Immerse in the acid solution a bright strip of Cu, a grey film is slowly deposited, which becomes brilliant white when it is rubbed.
<i>Presence of Hg''</i> (Mercuricum).
<i>Caution.</i>—A salt of Hg' may have been converted into an Hg'' salt during solution in HNO_3. The original substance should therefore be treated with AmHO, if it blackens Hg' was present, if not, Hg'' was originally in the substance.</td><td>Pour another portion of the original solution into much cold distilled water; a white precipitate or milkiness — <i>Presence of Bi</i>.
<i>Note.</i>—If Bi is suspected and no milkiness appears, add excess of AmHO to the liquid; Bi if present falls as hydrate; filter, pour one or two drops of boiling HCl upon the precipitate, and let them drop through into large excess of water; a milkiness on stirring shows <i>presence of Bi</i>.</td><td>To a portion add H_2SO_4 a white precipitate — <i>Presence of Pb</i>.
The presence of Pb should be confirmed by adding to a part of the original solution AmHO in excess, then $H\bar{A}$ in excess, then K_2CrO_4 a yellow precipitate shows <i>presence of Pb</i>.</td></tr>
</table>

<table>
<tr><td colspan="3">2 <i>The precipitate is yellow</i>: it may consist of CdS, As_2S_3, SnS_2.
Boil the precipitate with NaHO in excess, after decanting the liquid if possible:—</td></tr>
<tr><td rowspan="2"><i>The precipitate does not dissolve</i>: it is bright yellow and pulverulent— <i>Presence of Cd</i>.
The presence of Cd may be confirmed by showing that the precipitate produced by H_2S dissolves on being boiled with dilute H_2SO_4.</td><td colspan="2"><i>The precipitate dissolves</i>: it may consist of As_2S_3 or SnS_2; examine portions of the original solution as directed below:—</td></tr>
<tr><td>Acidify a portion with HCl, and place in it a piece of Zn which is known to be free from Sn. Sn is deposited on the Zn — <i>Presence of</i> Sn^{iv} (Stannicum).
The deposit of Sn should be detached from the Zn, and dissolved by boiling it in a test-tube with a little strong HCl, then $HgCl_2$ added; a white precipitate confirms the <i>presence of</i> Sn^{iv}.</td><td>Acidify a portion with strong HCl, immerse in it some pieces of bright Cu and boil, a black film is deposited on the Cu:— <i>Presence of As</i>.
<i>Note.</i>—If the yellow precipitate formed by H_2S appeared at once in the cold, the As is present as <i>Arsenosum</i>; if it formed only on boiling, <i>Arsenicum</i> is present.</td></tr>
</table>

3. *The precipitate is orange red*: it may consist of Sb_2S_3.

Confirm by acidifying a portion of the original solution with HCl, and immersing in it a piece of Zn resting on a slip of platinum foil, a black stain on the platinum (329):—
Presence of Sb.

Note.—**It occasionally happens, if the solution has not been sufficiently diluted, that H_2S gives a reddish precipitate with Pb-solution; such a precipitate, however, becomes *black* on adding more H_2S-water.**

TABLE III.A.—IRON GROUP.

949. The colour of the precipitate, produced by addition of AmCl and AmHO, will usually indicate whether it contains Fe, Al, Cr; the following tests may be made in confirmation.

To a portion of the original solution add KHO gradually until a precipitate has formed (see *Note* below Table), then add KHO in excess and stir well, one of the following results will be obtained :—

1 A *colourless* gelatinous precipitate is produced which dissolves in excess of KHO to a *colourless* solution the precipitate reappears on the addition of much AmCl to the KHO solution :— *Presence of* ***Al.***	2 A *pale green* gelatinous precipitate forms, which dissolves in excess of KHO to a *green* solution; when PbO_2 is added to this green solution and the liquid is boiled and then acidified with $H\bar{A}$, a yellow precipitate is produced.— *Presence of Cr*	3. A *reddish brown* precipitate forms, insoluble in excess of KHO Add KCyS to the original HCl solution, a blood-red coloration — *Presence of* Fe'''.	4. A *dingy green* precipitate forms, insoluble in excess of KHO, the precipitate if filtered off and exposed to the air gradually becomes brown — *Presence of* Fe''.

Note.—if a colourless gelatinous precipitate has been produced by AmCl and AmHO, and is not produced by addition of KHO, a silicate is probably present. Its presence is confirmed by fusing the solid substance in a bead of $NaAmHPO_4$ (690); or by evaporating the acid solution to dryness, a residue insoluble in acid is SiO_2 and may be tested by the bead as directed above.

TABLE III.B.—ZINC GROUP.

950. The colour of the precipitate, produced by addition of Am_2S or of H_2S to the alkaline solution, will distinguish Zn or Mn from Ni, Co, and Fe, and will usually distinguish Zn and Mn also from one another. See *a* and *b* below :—

a. If the precipitate is light in colour, add to some of the original solution KHO gradually until a precipitate forms, then add KHO in excess. A *white* precipitate which dissolves in excess of KHO and is reprecipitated by the addition of H_2S, proves the *presence of Zn.* A *white or brownish* precipitate, which is insoluble in excess of KHO and gradually turns dark brown when it is filtered off and exposed to the air, shows the *presence of Mn.*

b. If the precipitate is black it will consist of either CoS or NiS or possibly of FeS ; the metals Ni and Co may be distinguished from one another by the colour of their solutions—Ni being *green*, Co *pink*—and by the colours which they impart to the borax-bead (936, IV.). FeS is only precipitated here from a *dilute* solution of a ferrous salt : Fe'' is best detected by paragraph 949.

TABLE IV.—BARIUM GROUP.

951. Dip a loop of platinum wire either into the original solution, or into the HCl-solution of the precipitate which was produced by Am_2CO_3, and hold the wire in the Bunsen-flame. If the flame-coloration thus obtained is not decisive, the confirmatory tests which are placed at the foot of each column should be tried.

A yellowish green flame:—Presence of Ba	*A crimson flame,* appearing deep red through the indigo-prism — *Presence of Sr.*	*A red flame,* appearing dingy green through the indigo-prism — *Presence of Ca.*
Confirmatory -Add $CaSO_4$ to a perfectly cold portion of the HCl solution, a precipitate forms *immediately.*	*Confirmatory* -Add $CaSO_4$ to a perfectly cold portion of the HCl solution, no precipitate is produced, but a precipitate appears immediately on boiling	*Confirmatory* -Add $CaSO_4$ to part of the HCl solution and boil; no precipitate To the rest add AmHO in excess, and $Am_2C_2O_4$: a white precipitate forms.
Note — A green flame coloration may be produced by H_3BO_3.		

EXAMINATION FOR THE ACID-RADICLE.

952. If the acid-radicle has not been found already by the Preliminary Examination (938-941), or during the Examination for the Metal (942-946) or elsewhere, it must be tested for as is directed below.

Since the solubility of the substance (935) and the metal which it contains are now known, much trouble is usually saved by referring to the "Table of Solubilities" (1038).

For the following tests separate portions of the original substance must be used, and the tests must be tried in succession until an acid-radicle is found.

953. **Nitrate,** if it is present in small quantity only, may have escaped detection in the Preliminary Examination. It is well, therefore, to test for nitrate in the aqueous solution of the substance by ferrous sulphate solution and H_2SO_4 (537).

954. **Sulphate** —Acidify the liquid, or if the substance is a solid dissolve it, with dilute HCl, and add $BaCl_2$. If Pb, Hg′, or Ag has been found, acidify with HNO_3 and add $Ba(NO_3)_2$, since HCl and chloride would precipitate any of the above metals as chloride. A white precipitate forms, which does not disappear when the liquid is boiled.—*Presence of Sulphate.*

955. **$AgNO_3$ test.**—Acidify the liquid, or if the substance is a solid dissolve it, with dilute HNO_3· filter off any precipitate or residue which may form, and add $AgNO_3$-solution. A precipitate forms, which does not disappear when the liquid is warmed. Note the colour of the precipitate, and examine it according to the directions in 1, 2, or 3 below.—

1. *The precipitate is white;* it may consist of AgCl, AgCy, Ag_4FeCy_6, or AgCyS Make a portion of the original solution just acid with HCl, then add Fe_2Cl_6 —

A deep blue precipitate: *Presence of a ferrocyanide.*	A blood-red coloration· *Presence of a sulphocyanide.*

If Fe_2Cl_6 has yielded neither a precipitate nor coloration, indicating absence of a ferro- and sulpho-cyanide, decant the liquid from the precipitate caused by $AgNO_3$, pour upon the precipitate some strong HNO_3 and boil

The precipitate remains undissolved *Presence of a chloride.*	The precipitate dissolves: *Presence of a cyanide* Confirm the presence of a cyanide in a portion of the original solution by boiling it with $FeSO_4$, Fe_2Cl_6, and KHO, and acidifying with HCl: a blue precipitate *Presence of a cyanide.*

2 *The precipitate is yellow,* and may consist of AgBr or AgI. Test some of the original solution for an iodide by adding to it starch solution and KNO_2 solution, then acidifying (if not already acid) with HCl: a deep blue coloration:

Presence of an iodide.

If an iodide is not thus detected, add to another portion of the original solution several drops of Cl-water, if the liquid becomes yellow, and on being shaken with CS_2 colours that liquid reddish brown, it shows:

Presence of a bromide.

3. *The precipitate is orange red,* and is probably $Ag_6Fe_2Cy_{12}$. Make a portion of the original solution just acid with HCl, then add $FeSO_4$ solution; a deep blue precipitate shows:

Presence of a ferricyanide.

Note.—If Hg has been found, $HgCy_2$ may be present even if no precipitate has been formed on the addition of $AgNO_3$. If $HgCy_2$ has not been already detected in paragraph 936, and its presence is suspected, refer to paragraph 651.

956. **Phosphate, Arsenate.**—Acidify the liquid, or if the substance is a solid dissolve it, with dilute HNO_3. Filter if necessary, add a small quantity of this acid solution to some $AmHMoO_4$-solution, stir well and warm gently:—

A yellow precipitate shows:— *Presence of Phosphate.*	*If no precipitate forms,* heat to boiling, the formation of a yellow precipitate shows:— *Presence of Arsenate*

The presence of phosphate or arsenate may be confirmed by separating the yellow precipitate by filtration or decantation, and dissolving it by means of a little AmHO. When AmCl and $MgSO_4$ are added to this solution a crystalline precipitate will form. This is filtered off and washed with a little cold water, and several drops of $AgNO_3$-solution are poured upon it on the filter. If the precipitate becomes *yellow,* phosphate is present; if it becomes *brown,* arsenate is present. The presence of arsenate is further confirmed by the appearance of a yellow precipitate (As_2S_3), when H_2S is passed into the boiling HCl-solution of the original substance (313).

957. **Borate.**—Acidify the original liquid, or if the substance is a solid stir it, with HCl. Dip into this solution a piece of turmeric-paper, and dry it at a gentle heat. If the paper is *reddish-brown* when it is dry, and the colour changes to *dingy brown* or *blue black* when the paper is moistened with AmHO, the *Presence of Borate* is proved.

958. **Tartrate, Oxalate, Fluoride.**—The Acid-radicles, *one* of which may be present, are $\bar{T}$, C_2O_4, and F. If the metal already found belongs to Group I., II., III., or IV., the examination for one of these radicles must be commenced at paragraph 960; if the metal present is K, Na, or NH_4, examine by paragraph 959.

959. Make the solution just alkaline with AmHO, and add $CaCl_2$-solution in some quantity; then shake the liquid well and let it stand if no precipitate forms at once.

The Preliminary Examination (939, 940), will have indicated already which of these acid-radicles is present: it is therefore possible to decide which of the three columns below must be used for the further examination of the precipitate:—

I. *A crystalline precipitate, appearing after some time,* shows probable *presence of a tartrate.* Confirm by 1 or 2 below :— 1. Decant the liquid from the precipitate, add a little water and a single drop of AmHO, drop in a small crystal of $AgNO_3$ and warm very carefully; a mirror is formed below the crystal, *presence of a tartrate.* This test may be tried also with the original solution by pars 693 694 2. On acidifying some of the neutral original solution with $H\bar{A}$ and adding alcohol and $K\bar{A}$ (unless K or NH_4 has been detected in the substance), and stirring or shaking well, the formation of a crystalline precipitate shows *presence of a tartrate*	II. *A white pulverulent precipitate* shows probable *presence of an oxalate.* The presence of an oxalate, already indicated by the test in 939, 5, must be confirmed either by warming some of the original substance with MnO_2 and H_2SO_4, and testing for CO_2 gas (678): or by the more tedious method of filtering off and drying some of the above precipitate of CaC_2O_4, then igniting it and testing for a carbonate with HCl (677)	III. *A gelatinous flocculent precipitate* shows probable *presence of a fluoride.* Confirm by filtering and warming some of the precipitate, or of the substance if solid, in a small cup of lead or platinum with strong H_2SO_4; fumes will be evolved which etch a properly prepared watch glass (606).

960. An oxalate or fluoride of Ba, Sr, Ca, or Mg will have been already detected. A tartrate of any one of these metals is found by gently heating the finely powdered substance if it is solid, or the neutral solution if it is liquid, with *very dilute* AmHO and a crystal of $AgNO_3$: the formation of a mirror shows the *presence of a tartrate.* If a metal of Group I., II., or III. has been found, the metal must be separated before the acid-radicle is tested for.

If the metal belongs to Group I. or II., make the original solution just acid with HCl, and either add H_2S-solution, or pass H_2S into the liquid for about five minutes. Filter off the precipitate and add more H_2S to the filtrate; if no more precipitate forms,* boil the liquid in a porcelain dish until it ceases to smell of H_2S, and examine the liquid by par. 959.

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* If any further precipitate forms, add more of the reagent, filter, and test again by adding more of the reagent; this addition of reagent, filtration, and testing the filtrate is repeated until no further precipitate forms when the reagent is added again to the filtrate.

If the metal belongs to Group III., add AmCl, and AmHO till the liquid is alkaline; then add Am_2S, boil and filter; now add more Am_2S to the filtrate, and if no more precipitate forms (see Footnote, page 318), add $H\bar{A}$ in excess, boil, filter off S if necessary, and examine the liquid by paragraph 959.

961. **If no Acid-radicle can be found in any quantity** and the substance is not metallic in appearance, it is probably an oxide, hydroxide, or perhaps a sulphide.

Most sulphides yield sulphate when they are boiled with strong HNO_3· the sulphate may be detected, after the acid has been diluted, by paragraph 954.

An oxide may often be known by its colour (932), or by some characteristic test. Thus black MnO_2 may be identified by evolving Cl when it is heated with HCl, and red Pb_3O_4 by leaving brown PbO_2 when it is treated with dilute HNO_3.

EXAMINATION OF GROUP III.A.

962. **The Precipitate in Group III.A.**, which has been produced by the addition of AmCl and AmHO to the original acid solution of a substance which is insoluble in water, may consist of $Al_2H_6O_6$, $Fe_2H_6O_6$, $Cr_2H_6O_6$; or of Fe, Al, Cr, Zn, Mn, Ni, Co, Ba, Sr, Ca, Mg as phosphate or oxalate; or of Ba, Sr, Ca as chromate or fluoride.

The Systematic Examination of a precipitate in Group III.A. for these substances is usually only necessary when a phosphate has been detected (942), and occasionally when an oxalate (939, 5), a chromate (946), or a fluoride (940) is present.

If a precipitate forms in Group III.A., and the above acid-radicles are absent, examine it by paragraph 949.

If one of the above acid-radicles is present, the metal must be tested for as is directed below, unless it has been already detected with certainty during the Preliminary Examination, or by the ordinary procedure given in the General Table (942).

963. **Note the Appearance of the Precipitate** which has been produced by AmCl and AmHO: then add Am_2S to

the liquid and precipitate, and stir well. According to the appearance which the precipitate now presents, proceed to examine further by paragraph 964 or 965.

964. *The Precipitate, after the Addition of Am_2S, is Black.* —It may consist of FeS, CoS, or NiS. The means of distinguishing these three metals is given below. Note first the colour of the original solution (937, I.).

<table>
<tr>
<td rowspan="2">a. The colour of the original solution was yellow or reddish-brown —
Presence of Fe'''.
(Ferricum.)
This is confirmed by the precipitate produced by AmCl and AmHO in the original solution having been pale-yellow or reddish-brown, and becoming black on addition of Am_2S
As a confirmatory test add to a portion of the original solution, acidified with HCl if necessary, a few drops of KCyS solution, a blood-red coloration is produced.</td>
<td rowspan="2">b. The colour of the original solution was pink, or possibly blue becoming pink on dilution
Presence of Co.
The presence of Co may be confirmed by adding excess of KHO to the original solution, a blue precipitate turning red on boiling shows the presence of Co.
Co will also have been detected by its borax bead (936, IV.)</td>
<td colspan="2">c. The colour of the original solution was green —Presence of Ni or Fe''.
Note the effect which was produced by the addition of AmCl and AmHO —</td>
</tr>
<tr>
<td>A blue solution was produced — Presence of Ni
The presence of Ni may be confirmed by adding excess of KHO to a part of the original solution, a light green precipitate shows the presence of Ni.
Ni will also have been detected by its borax bead (936, IV)</td>
<td>A dingy green precipitate, which, when shaken round in a porcelain dish and left for several minutes exposed to the air, becomes brown — Presence of Fe''.
(Ferrosum.)
Confirm by adding K_3FeCy_6 to some of the original solution, acidified with HCl if necessary; a dark blue precipitate is produced.</td>
</tr>
</table>

965. *The Precipitate, after the Addition of Am_2S, is White or Light-coloured.*—It may consist of $Cr_2(HO)_6$, $Al_2(HO)_6$, ZnS, MnS; [or of $BaCrO_4$; or of Ba, Sr, Ca, Mg, as phosphate, oxalate, fluoride, silicate, or possibly borate]. The substances in brackets can, however, only be present if the liquid given for analysis was acid in reaction, or if a solid was given which was insoluble in water but soluble in acid.

To a small portion of the original solution add KHO drop by drop until a precipitate is produced. (See Note below the Table.) Observe the colour and appearance of this precipitate, then add a larger quantity of KHO and stir or shake the liquid well. One of the following results (*a* or *b*) will be noticed :—

a. *The precipitate at first produced by KHO re-dissolves when the KHO is added in larger quantity;* indicating the presence of Al, Zn, or Cr. Note the appearance of the precipitate:—

A pale green flocculent precipitate — *Presence of Cr.* Confirm by boiling some of the green KHO solution with lead-peroxide (PbO_2), a yellow liquid is obtained, which, if decanted, yields a yellow precipitate on addition of $H\bar{A}$ in excess The colour of the solution and of the borax bead will also have detected Cr. *Note.*—If the Cr was present in the acid-radicle of a chromate, AmCl and AmHO will have yielded no precipitate, but there will have been a green precipitate on addition of Am_2S and heating	***A white precipitate, which is re-precipitated from the KHO solution,*** by addition of a few drops of H_2S-water but is not re-precipitated by addition of AmCl: *Presence of Zn* Confirmed by no precipitate having been produced by AmCl and AmHO, but a white precipitate by addition of Am_2S, or of H_2S after AmHO	***A white flocculent precipitate, not reprecipitated*** from the KHO solution by addition of a few drops of H_2S-water or by boiling, but reprecipitated immediately if sufficient AmCl solution is added — *Presence of Al* (see Note). *Note* —$AlPO_4$ is also precipitated here, and is soluble in KHO, but differs from $Al_2(HO)_6$ in being reprecipitated from the KHO solution by addition of excess of $H\bar{A}$; if PO_4 has been detected the analysis is finished. $AlPO_4$ is soluble only in acids, hence the original liquid must have been acid or the original solid substance insoluble in water.

Note.—Sometimes a flocculent precipitate is produced on adding AmCl and AmHO to the original solution, and yet no precipitate is produced by KHO; this renders probable the presence of an alkaline silicate. In this case evaporate a portion of the solution to dryness with HCl; warm the residue with HCl: if any insoluble residue is left, the *presence of a silicate* is proved; filter and test the filtrate for K or Na.

b. *The precipitate at first produced by KHO does not redissolve on addition of more KHO;* the precipitate will have one of the following appearances:—

A white or dingy yellow precipitate, rapidly darkening when shaken in the air.— *Presence of Mn.* *Confirmed* in the preliminary examination by producing a green mass when fused with Na_2CO_3 and KNO_3, and by no precipitate having been produced on addition of AmCl and AmHO, unless the solution was exposed for some time to the air.	***A pale yellow precipitate, not darkening in the air*** this precipitate will also have been produced by AmCl and AmHO and its colour will not have been altered by addition of Am_2S — *Presence of $BaCrO_4$.* Ba has been already detected by the flame coloration (936, II), and a chromate by the change of colour of the orange red liquid to green on adding H_2S (946).	***A white precipitate, not darkening in the air.—*** Presence of Ba, Sr, Ca, or Mg as phosphate, oxalate, &c. Pass on to par 966. *Note* —This precipitate can only be formed if the original solution was acid.

966. *The precipitate may consist of Ba, Sr, Ca, or Mg as phosphate, oxalate, borate,* fluoride, or silicate.*

Examine for the metal and acid-radicle as directed below, working through the columns from left to right, unless the presence of a phosphate (942), oxalate (936, I. 4, or 939, 5), borate (936, II., or 937, V., or 941), or fluoride (940), has been already proved, in which case proceed at once to examine according to 967, or by the suitable column below.

If this precipitate is proved to contain a phosphate, oxalate, borate, fluoride, or silicate, the further examination for an acid-radicle becomes unnecessary.

1. Add AmHO in excess to a portion of the original solution†, filter, dry the precipitate and heat a portion of it to a *dull red heat* for several minutes on platinum foil Place the foil in a test-tube and pour a few drops of dilute HCl upon it; if the residue dissolves with effervescence, it shows the *presence of an oxalate.* Test this HCl solution for Ba, Sr, Ca, Mg, by 942, using it as the original solution. If the ignited precipitate does not dissolve with effervescence, pass on to column 2.	2. Stir some of the precipitate produced by AmHO†, column 1, on a watch glass, with a little water to which a few drops of HCl have been added, until the precipitate is dissolved Dip into the liquid a slip of turmeric paper, and dry at steam heat, the slip appears *reddish-brown*, and becomes *blue-black* when moistened with AmHO — *Presence of a borate.* Examine for the metal present by 968 If a borate is not found, proceed to column 3.	3. Place another portion of the precipitate produced by AmHO†, column 1, in a leaden cup or platinum crucible, add strong H_2SO_4 and warm gently, covering the vessel with a watch glass, which has been coated with a film of wax and has had characters traced with a sharp point through the wax (606), the glass is etched — *Presence of a fluoride* Examine for the metal present by 968 If no fluoride is found, test for a silicate by par. 968.

967. *Examination of a phosphate.*—Add to a portion of the cold original solution AmHO drop by drop, whilst constantly shaking the liquid, until a slight precipitate is formed which does not disappear on stirring; then add $H\bar{A}$ and $Na\bar{A}$ solution, and drop in Fe_2Cl_6 until the liquid after being well shaken has a brown or red colour; boil and filter immediately. The precipitate is thrown away. To

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* Borates of Ba, Sr, and Ca, are rarely precipitated in Group III., since they are soluble in the AmCl formed on adding AmHO to the HCl solution.

† If a solid is being examined, a portion of the original solid is employed for this test, instead of the precipitate produced by AmHO in the HCl solution.

the filtrate AmCl is added, then AmHO in small excess, and the liquid is boiled and filtered : the liquid or filtrate, which must smell strongly of NH_3, is then tested for Ba, Sr, Ca, and Mg (942).

Note.—If any precipitate is formed by Am_2CO_3, it must be well washed several times with boiling water in order to prevent the coloration of Na from masking that of Ba, Sr, or Ca. This risk is avoided by substituting $Am\bar{A}$ for $Na\bar{A}$ in precipitating the phosphate radicle.

Explanation of the Method for Examination of a Phosphate.

In order to detect Ba, Sr, Ca, or Mg present as phosphate, the PO_4 **must be removed from the solution and the metal left as chloride. This** is effected by neutralising all HCl and leaving the solution acidified only by free $H\bar{A}$; by addition of Fe_2Cl_6 in slight excess and boiling, all PO_4 is precipitated as $FePO_4$ which though soluble in HCl is perfectly insoluble in $H\bar{A}$. On filtering, $FePO_4$ remains upon the filter, and the filtrate contains the metal (Ba, Sr, Ca, or Mg) as chloride together with a little Fe, but is perfectly free from phosphate.

968. *Examination of a borate, fluoride, or silicate.*—A portion of the original substance is placed in an evaporating basin, which is then nearly filled with dilute HCl, and the liquid is entirely boiled away ; the dish is once more filled with acid, and again evaporated to dryness : the residue in the dish is then warmed with dilute HCl, and filtered if any residue is left :—

Solution : the HCl solution is examined for Ba, Sr, Ca, Mg by 942, this solution being treated as is there directed for the original solution.	*Residue :* if any residue remains insoluble in HCl, it proves :— *Presence of a silicate.*

Explanation of the process.—The evaporation to dryness with HCl removes H_3BO_3 and HF, which volatilise, whilst SiO_2 is left insoluble in HCl and therefore remains as an insoluble powder on afterwards warming with HCl ; this residue of SiO_2 also remains, however, if HF was present, being caused by the corrosion of the dish ; since however HF if present has already been found, no notice will then be taken of the residue.

THE SUBSTANCE POSSESSES METALLIC LUSTRE.

969. This shows the probable absence of an acid-radicle. The only non-metallic elements likely to be present are free carbon as graphite, free iodine, or combined sulphur, since several sulphides are decidedly metallic in appearance*. The presence of the metals Ni, Co, Fe in the free state, and of Fe_3O_4, may be shown by the substance being attracted by a magnet. Pb, or graphite, will be recognised by marking paper when rubbed upon it, as a black lead pencil does: the mark due to Pb disappears if wetted with dilute HNO_3.

Preliminary Examination.

Experiment.	Observation	Inference.
I. Heat a portion of the substance in a small ignition-tube	1. A black lustrous mirror forms .	Presence of As
	2. A yellow sublimate forms, melting, when heated, to brown drops . .	Presence of S.
	3. Violet vapour is given off, condensing to black scales on the sides of the tube	Presence of I.
	If the substance sublimes entirely as violet vapour	Iodine alone is present.
II Heat a portion in the inner blowpipe flame in a cavity scooped on a piece of wood charcoal.	1. Smell of garlic . . .	Presence of As.
	2. If an incrustation forms, refer to par 936, V., where in the second and third column will be found the appearance of the incrustation and the metal whose presence it indicates.	
	3. Smell of burning sulphur . .	Presence of S.
	4. A green flame coloration . .	Presence of Cu.
III. Place a portion of the substance in a piece of hard glass tube open at both ends, and heat it strongly in the Bunsen flame, and afterwards in the blowpipe flame if necessary.	1. A gas is evolved from the upper end which smells of burning sulphur and turns a piece of filter-paper moistened with $K_2Cr_2O_7$ solution green	Presence of S.
	2. The substance burns completely but slowly away, glowing when strongly heated. This effect is best seen by heating it strongly on Pt-foil by directing the blowpipe flame on the under surface of the foil .	Presence of C.

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* The most common sulphides which possess metallic lustre are *copper pyrites* and *iron pyrites*, which resemble brass in appearance: and *galena*, which resembles lead.

After the Preliminary Examination has been completed, proceed as is directed in paragraph 935.

Sulphide, Phosphide, Arsenide.—No acid-radicle need be tested for except S, P and As, which are most easily detected after they have been converted into acids by oxidation. A portion of the finely-powdered substance is accordingly dissolved in strong HNO_3, and this solution is diluted with water: H_2SO_4 is then tested for by $BaCl_2$, and H_3PO_4 and H_3AsO_4 by $AmHMoO_4$ (956).

THE SUBSTANCE IS INSOLUBLE BOTH IN WATER AND IN ACIDS.

970. **An Insoluble Substance may be** any one of the following. Those included in brackets are less likely to occur than the others, since they dissolve when they are heated with sufficient water or acid. As will be seen below, the colour frequently serves to indicate what substance is present. For a fuller description see paragraph 1074.

1. *White:* $BaSO_4$, $SrSO_4$, ($CaSO_4$), $PbSO_4$, ($PbCl_2$), AgCl,* SiO_2, ignited Al_2O_3, SnO_2, Sb_2O_5, Sb_2O_4, CaF_2.
2. *Dark-coloured:* ignited Fe_2O_3 (dark brown or black), ignited Cr_2O_3 (dark green or black), $FeCr_2O_4$ and ignited $PbCrO_4$ (dark brown), C (black).
3. *Yellow:* S, AgBr,* AgI,* (PbI_2).
4. An insoluble silicate may also be present.

Examine the substance by paragraph 971, 973, or 974.

971. **The Substance is White** (970, 1).—If sufficient substance is available, try the following preliminary tests (I.–IV.); if not, proceed at once to paragraph 972.

1. Pour a drop of Am_2S upon a small portion of the substance. If it blackens, the presence of Pb or Ag is probable; if it becomes yellow or orange-red, the presence of Sn or Sb is probable. Pass on to Test IV. in the following Table.

If no change of colour is produced by Am_2S, try Tests II. and III. in the following Table.

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* Darkens quickly in sunlight, and slowly in ordinary daylight.

Experiment.	Observation	Inference.
II Moisten a clean loop of platinum wire, immerse it in the powdered substance, and heat the adhering powder for a short time strongly in the *inner* blowpipe flame; moisten the loop with a drop of strong HCl, and hold it in the Bunsen flame	A *yellowish green* flame.	*Presence of Ba* Test for SO_4 by Exp III.
	A *crimson* flame, appearing *deep red* through the indigo-prism.	*Presence of Sr*
	A *reddish* flame, appearing *dusky green* through the indigo prism.	*Presence of Ca*
III Fuse a portion of the substance, mixed with Na_2CO_3 in fine powder, in a cavity scooped upon a piece of wood charcoal, in the inner blowpipe flame *produced from a spirit lamp flame*	The cooled mass, if detached from the charcoal, placed upon a bright silver coin, and moistened with water, gives, when crushed with the blade of a knife, a black stain	*Presence of a sulphate.* *Note*.—If SO_4 is not found, test for F by 940 or 606, and further examine the substance by 972.
IV Mix some of the finely powdered substance with powdered Na_2CO_3 and KCy, on a small cavity made in a piece of wood charcoal, and heat the mixture in the inner blowpipe flame. If metallic globules form, detach one and strike it smartly with the pestle on the inverted mortar. Also take a globule on the point of a penknife and try if it marks paper as black lead does.	White metallic *malleable* globules, which mark paper, and if dissolved in HNO_3 give a white precipitate with H_2SO_4; a yellow incrustation is also formed on the charcoal	*Presence of Pb* as chloride, sulphate, or chromate. Refer to the *Note* below this Table.
	White metallic *malleable* globules which do not mark paper; they dissolve in HCl, the solution giving a white precipitate with $HgCl_2$.	*Presence of SnO_2*.
	White metallic *brittle* globules, which, if dissolved in boiling HCl, give an orange red precipitate with H_2S; white incrustation.	*Presence of Sb_2O_4 or Sb_2O_5*
	White scales, no incrustation: the metal is insoluble in HCl, and if dissolved in HNO_3 gives no precipitate with H_2SO_4 but a white precipitate with HCl; the original substance is instantly blackened by a drop of Am_2S, and is dissolved on being warmed with AmHO.	*Presence of AgCl.* Proceed to 972

Note.—Boil some of the substance with much water, add HNO_3 and $AgNO_3$, a white precipitate shows *presence of $PbCl_2$*.

Test for chromate by fusing in a porcelain crucible with $Na_2CO_3 + K_2CO_3$; a yellow mass on cooling shows *presence of $PbCrO_4$*.

The presence of $PbSO_4$ may be proved by Exp. III., and by pouring upon the original substance $H_2\overline{T}$ then excess of AmHO, and boiling:

then filtering if necessary. acidifying a portion of the clear solution with $H\bar{A}$, and adding K_2CrO_4; a yellow precipitate shows the *presence of Pb.* Another portion may then be acidified with HCl, and if $BaCl_2$ produces in it a white precipitate insoluble on boiling, *presence of SO_4* is proved.

If none of the above substances have been detected, test for Al_2O_3 and SiO_2 by heating the substance strongly on charcoal in the outer blowpipe-flame, after moistening it with $Co(NO_3)_2$-solution; a blue mass shows *Presence of Al_2O_3*, unless SiO_2 is found by the next test.

Fuse some of the substance in a clear bead of $NaAmHPO_4$; particles float undissolved in the bead:—*Presence of SiO_2, or silicate.* Proceed to par. **972.**

972. Mix a portion of the finely-powdered substance with four or five times as much fusion mixture ($Na_2CO_3 + K_2CO_3$) on a piece of platinum foil, or in a porcelain crucible if Pb or Ag is present. Heat the mixture for several minutes in the blowpipe-flame (see fig. 80, **1076**), keeping the alkaline carbonates in a fused and liquid condition. Then boil the cool mass with water, and filter the liquid: wash the residue well on the filter, and examine the residue and the filtrate as is directed below.

(*a*) *Remove the Residue* from the filter and heat it with dilute HNO_3. If it does not dissolve, decant the acid, and heat the residue with a small quantity of a mixture of dilute HNO_3 and HCl. If the residue is still undissolved, fuse it again with fresh fusion mixture, and treat it as is directed above.

Examine the acid solution for the metal by par. **942**

(*b*) *Examination of the Filtrate.*—When the metal has been detected, it will usually be possible to limit the number of acid-radicles which need be tested for, by referring to the list in par. **970.** Tests are given below for all the acid-radicles which are likely to occur in an insoluble substance. A separate portion of the filtrate must be used for each test.

Sulphate: acidify a portion with HCl and add $BaCl_2$: a white precipitate will form.

Chloride: acidify a portion with HNO_3 and add $AgNO_3$: a white precipitate will be produced, which is easily soluble in AmHO.

Bromide, Iodide: if the precipitate produced by $AgNO_3$ has a yellow tinge and is not easily soluble in AmHO, add to a fresh portion of the filtrate a little CS_2; then add acidified $CaCl_2O$-solution drop by drop, shaking well between each addition. A brown coloration of the CS_2 shows a *bromide*, a violet coloration an *iodide.*

Silicate: make a portion acid with HCl and evaporate to perfect dryness; if an insoluble residue is left when the solid matter in the dish is warmed with dilute HCl, this proves the presence of silica or a silicate.

Fluoride: acidify with $H\bar{A}$ and add $CaCl_2$: a gelatinous white precipitate will form. Confirm by trying the tests in paragraphs 940 and 606 on some of the original substance.

973. The Substance is Dark-coloured (970, 2).

Place some of the substance on a piece of platinum foil, and heat the foil strongly on its under surface by the blowpipe-flame :—

The substance burns away slowly but completely:— *Presence of C.*	*The substance does not burn away;* place upon it three or four times as much powdered Na_2CO_3 and KNO_3, and fuse for some time:—
	1. The substance dissolves, forming a yellow mass when cold:—*Presence of Cr_2O_3.*
	2. The substance remains undissolved as a dark brown powder, and the mass on cooling is white:—*Presence of Fe_2O_3.** Confirm by boiling the mass with water, filtering, and fusing the brown residue into a borax bead (200).

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* $FeCr_2O_4$ shows both reactions 1 and 2, since it contains both Fe and Cr.

974. The Substance is Yellow (970, 3).

Heat a portion of it strongly in a small ignition-tube; one of the following results will occur:—

It will fuse and sublime, the sublimate being yellow and melting to reddish-brown drops when it is heated:—	*It will fuse but not sublime.*
Presence of S. *Note*.—If the substance sublimes entirely it consists only of sulphur.	Examine a portion of the substance for Ag and Pb and for Br and I by paragraph **972**.

ANALYSES OF SIMPLE SALTS, SHOWING HOW TO ENTER RESULTS.

A. *Liquid given for analysis.*

Preliminary Examination for the Metal.

Experiment.	Observation.	Inference.
1. Noted the colour of the liquid.	Blue.	Presence of Cu.
2. Dipped into it a piece of blue litmus-paper.	The paper was turned red, but on addition of Na_2CO_3 no effervescence occurred	Presence of a salt with acid reaction.
3. Evaporated a few drops slowly upon a watch glass.	Pale blue residue left	Presence of some dissolved solid.
Ignited the residue strongly.	Blackened without smell of burning	Absence of organic acid-radicles.
4. Added KHO solution and boiled.	No smell of NH_3	Absence of NH_4.
5. Dipped a loop of platinum wire into the solution and held it in the flame.	*Bright green* flame; on moistening the wire with strong HCl and again holding it in the flame it gave a blue coloration.	Presence of Cu.
6. Dipped a clear borax bead into a portion of the residue from 3, and fused in the outer and inner blowpipe flame.	In outer flame the bead was *green* whilst hot, *blue* when cold. In inner flame it became colourless.	Presence of Cu.

Preliminary Examination for the Acid-radicle.

Experiment.	Observation.	Inference.
1. Added dilute H_2SO_4 and warmed.	No gas was evolved.	Absence of carbonate, sulphite, sulphide, and nitrite.
2. Added strong H_2SO_4 and warmed.	No gas was evolved And no red fumes on adding Cu clippings.	Absence of chlorate, &c. Absence of nitrate.

Examination for the Metal.

To a portion of the liquid added HCl:—

No precipitate. Absence of Group I.	Added strong H_2S-water:—
	A brownish black precipitate. Presence of Sn'', Hg'', Bi, Pb, or Cu. Allowed the precipitate to settle, poured off as much of the liquid as possible and boiled the precipitate with excess of NaHO, it remained undissolved. Since the original solution was blue, a clean knife blade was dipped into a portion of it acidified with H_2SO_4, red copper was deposited on the steel:— *Presence of Cu.*

Examination for the Acid-radicle.

Added HCl and $BaCl_2$; a white precipitate was produced which did not disappear on boiling:—*Presence of sulphate.*

Found Cu, SO_4.

B. *Liquid given for analysis.*

Preliminary Examination for the Metal.

Experiment.	Observation.	Inference.
1. Noted the colour of the liquid.	Light yellow.	Presence of a neutral chromate.
2. Dipped red litmus-paper into the liquid.	It was turned *faintly* blue	Probable presence of an alkali-salt.
3. Evaporated a few drops to dryness upon a watch glass.	A yellow residue remained.	Presence of some dissolved solid.
Ignited the residue strongly.	No blackening.	Absence of organic acid-radicle.
4. Boiled a portion with excess of KHO.	No smell of NH_3	Absence of NH_4.
5. Dipped a loop of platinum wire into the solution, and held it in the Bunsen flame.	A pale lavender flame, appearing crimson through the indigo-prism.	Presence of K.
6. Dipped a clear borax-bead into the residue from 3, and fused in the outer and inner blowpipe flames.	Outer flame: *brown*, hot; *green*, cold. Inner flame: *green*, hot and cold. *Brown* colour not reappearing in the outer flame.	Presence of Cr as a chromate.

Examination for the Metal.

Since the liquid was alkaline in reaction, added to a small portion of it a few drops of HNO_3 until it became acid, this produced no precipitate.

To another portion added HCl, the liquid turned from yellow to orange red, indicating presence of a neutral chromate:—

<table>
<tr><td rowspan="4"><i>No precipitate.</i> Absence of Group I.</td><td colspan="4">Then added H_2S-water and warmed :</td></tr>
<tr><td rowspan="3">The liquid became green, and white S was deposited:
<i>Presence of a chromate.</i></td><td colspan="3">To another portion of the original solution added AmCl, then AmHO, no precipitate formed. then added Am_2S and boiled :</td></tr>
<tr><td rowspan="2"><i>No precipitate</i> in the cold, but on boiling a green flocculent precipitate gradually formed, evidently consisting of Cr_2Ho_6 reduced from the chromate.</td><td colspan="2">Added to another portion of the solution AmCl, AmHO, and Am_2CO_3 :</td></tr>
<tr><td><i>No precipitate.</i> Absence of Group IV.</td><td>Added to the same portion Na_2HPO_4 and stirred :
<i>No precipitate. Absence of Mg.</i></td></tr>
</table>

Since K was found by the flame coloration in the Preliminary Examination, its presence was confirmed by stirring a portion of the solution, to which a few drops of HCl had been added, with $PtCl_4$ on a watch glass; a yellow crystalline precipitate forming on the lines rubbed by the rod:—*Presence of K.*

Found K, CrO_4.

C. A *white crystalline substance given for analysis.*

Boiled a small portion with water; it dissolved completely.

Preliminary Examination for the Metal.

Experiment.	Observation.	Inference.
1. Heated in a small dry test-tube.	The substance gave off red fumes and oxygen gas, which inflamed a glowing splinter of wood. It left a yellow residue.	Presence of a nitrate of a heavy metal. Presence of Pb, Sn, or Bi.
2. Dipped a loop of moistened platinum wire into the powdered substance, moistened with strong HCl and held in the Bunsen flame	A pale blue flame.	Presence of As, Sb, Pb.
3. Heated a small portion in a cavity on wood charcoal in the inner blowpipe flame.	Deflagration occurred. A white malleable globule remained which easily marked paper. A yellow incrustation.	Presence of a chlorate or nitrate. Presence of Pb.
4. Fused on charcoal in the inner blowpipe flame with Na_2CO_3 and KCy.	Same result as with 3.	Presence of Pb.

Examination for the Metal in Solution.

Boiled a portion of the powdered substance with water, cooled, added HCl to a part of the clear solution, a white precipitate was formed, which disappeared on boiling, but appeared again in the crystalline form on cooling the liquid (*Probable Presence of Pb*).

Confirmed the presence of Pb by decanting the liquid and boiling the precipitate with AmHO, it remained white and did not dissolve; decanted the ammoniacal liquid, dissolved the precipitate in a little $H\bar{A}$, and added K_2CrO_4, a yellow precipitate.—*Presence of Pb*.

Since a nitrate was found in the Preliminary Examination, its presence was confirmed by adding strong H_2SO_4 to the aqueous solution of the substance, and pouring solution of $FeSO_4$ carefully upon the cooled liquid in a test-tube; a brown layer formed on the surface of the acid:—*Presence of* NO_3.

Also some of the solution, when heated with Cu and strong H_2SO_4, evolved reddish brown fumes:—*Presence of* NO_3.

Found:—Pb, NO_3.

D. *A white powder, emitting no smell, given for analysis.*

Boiled some of the powdered substance with water, it did not dissolve: added a few drops of strong HCl and boiled, the substance dissolved completely (absence of Group I.) without effervescence.

Preliminary Examination for the Metal.

Experiment.	Observation.	Inference.
1. Heated in a small dry test-tube.	No change.	**Absence of volatile and fusible substances and of water of crystallisation, &c.**
2. Dipped a moistened loop of platinum wire into the powdered substance, moistened the powder with strong HCl and held it in the Bunsen flame.	**No flame coloration.**	**Probable absence of K, Na, Ba, Sr, Ca, Cu, &c.**
3. Heated a portion of the powder strongly on charcoal in the inner blowpipe flame.	**The substance did not fuse. A white luminous residue, which when moistened on red litmus-paper showed an alkaline reaction.**	**Absence of alkali salt. Presence of Ba, Sr, Ca, Mg.**

Examination of the Solution.

To a portion of the HCl solution added H_2S-water; no precipitate:—*Absence of Group II.*

To another portion of the HCl solution added AmCl and AmHO in excess; a white flocculent precipitate. Then added Am_2S, the colour of the precipitate was unchanged.

To a few drops of the HCl solution added KHO drop by drop, until a white flocculent precipitate formed; then added excess of KHO and boiled, the precipitate did not dissolve, and it did not become discoloured in the air:—*Presence of Ba, Sr, Ca, Mg as phosphate, oxalate, &c.*

Added a drop of the original HCl solution to some $AmHMoO_4$-solution in a test-tube, and warmed gently; a yellow precipitate:—*Presence of PO_4.*

To another portion of the *cold* HCl solution added AmHO drop by drop whilst constantly stirring, until a precipitate formed which did not redissolve; then added $H\bar{A}$ and $Na\bar{A}$-solution until this precipitate dissolved when the liquid was well shaken. Fe_2Cl_6 was added until the liquid appeared red, it was then boiled and immediately filtered. To the filtrate AmHO was added in excess and the precipitate filtered off, then Am_2CO_3 was added; a white precipitate formed:—*Presence of Ba, Sr, or Ca.*

Filtered, washed, and dissolved the precipitate by pouring upon it a few drops of boiling HCl. Dipped a loop of platinum wire into the solution, and held it in the Bunsen-flame; a red flame appearing dingy green through the indigo prism:—*Presence of Ca.*

A few drops of the solution, when they were boiled with $CaSO_4$, gave no precipitate, proving the absence of Ba and Sr. The presence of Ca was therefore confirmed by adding to the rest of the solution excess of AmHO and then $Am_2C_2O_4$, when a precipitate was formed.

Found Ca, PO_4.

E. *A white odourless powder given for analysis.*

Boiled a portion of the substance with water, it did not dissolve. Added HCl and heated again, it dissolved completely (absence of Group I.) and without effervescence.

Preliminary Examination.

Experiment.	Observation.	Inference.
1. Heated a portion in a small dry test-tube.	The substance blackened slightly; it evolved CO_2 which turned a drop of lime-water milky. The residue in the test-tube effervesced with HCl whereas the original substance did not.	Presence of an organic salt of K, Na, Ba, Sr, Ca, or Mg.
2. Held a portion of the substance on a loop of platinum wire in the Bunsen flame; then moistened with HCl and again held in the flame.	A *red* coloration, appearing *dusky green* through the indigo-prism.	Presence of Ca.
3. Heated on charcoal in inner blowpipe flame.	A white luminous mass remained, which when moistened on red litmus paper turned it blue.	Presence of Ba, Sr, Ca, or Mg.

Examination for the Metal.

Added to a portion of the HCl solution strong H_2S-water.

No precipitate:— Absence of Group II.	To another portion of the HCl solution added AmCl then AmHO in excess, *a white precipitate formed;* added Am_2S, the colour of the precipitate remained unaltered. To another portion of the solution added KHO drop by drop until a precipitate formed, then more KHO and boiled; a white precipitate insoluble in KHO, and not darkening in the air; *—Presence of oxalate, phosphate, &c., of alkaline earths* Since the presence of an oxalate of an alkali- or alkaline earth-metal was indicated in the Preliminary Examination, a portion of the original substance was heated on platinum foil, then dissolved in HCl (effervescence) and to the solution excess of AmHO and of Am_2CO_3 was added. The liquid was warmed and put aside till the precipitate subsided, then the liquid was decanted and the precipitate dissolved in a few drops of HCl. A platinum wire dipped into this HCl solution gave a *red* flame coloration, appearing *dusky-green* through the indigo-prism:—*Presence of Ca.* [Confirmed by another portion of this HCl solution giving a white precipitate with excess of AmHO and $Am_2C_2O_4$.]

The presence of C_2O_4 was confirmed by heating some of the original substance with strong H_2SO_4 : gases were evolved which rendered milky a drop of lime-water and burnt with a blue flame, showing presence of CO_2 and CO :—*Presence of* C_2O_4.

Found Ca, C_2O_4.

F. *A yellow metallic-looking substance given for analysis.*

Preliminary Examination.

Experiment.	Observation.	Inference.
1. Heated a portion of the substance strongly in a small tube closed at one end.	A yellow sublimate formed which melted into brown drops a smell of burning S was evolved, and a piece of paper dipped into $K_2Cr_2O_7$ solution when placed in the mouth of the tube became green. A brown residue left	Presence of S. Probable presence of Fe.
2. Heated strongly on charcoal in the inner blowpipe flame.	Strong smell of burning S The residue fused into a dark-coloured globule, which when cold was strongly attracted by the magnet.	Presence of S. Probable presence of Fe
3. Heated a fragment strongly in a glass tube open at both ends, and held obliquely in the flame.	A gas was evolved which had a suffocating smell, and which turned $K_2Cr_2O_7$ paper green.	SO_2 from combustion of S

Solution of substance.

Warmed the powdered substance with a mixture of strong HNO_3 and HCl, it dissolved completely; boiled down in an evaporating dish, adding strong HCl several times; when nearly dry, diluted with water.

Examination of the solution for the Metal.

Since the substance dissolved entirely in the presence of HCl, Group I. is absent; to a portion of the solution added H_2S-water :—

The solution became colourless, and white S was precipitated :—*Probable presence of a ferric salt.*	To another part of the solution added AmCl, then excess of AmHO, a brown flocculent precipitate formed : to a fresh part added excess of KHO, brown flocculent precipitate insoluble in KHO, *Presence of Fe'''.* Confirmed the presence of Fe by adding to a fresh part of the original solution KCyS solution, *a blood-red solution: Presence of Fe'''.*

Confirmed the presence of S by adding to a part of the original solution $BaCl_2$, a white precipitate formed which did not disappear on boiling :—*Presence of SO_4 derived from oxidation of sulphur.*

Found Fe, S.

G. *A white powder given for analysis.*

Boiled a small portion with water, it did not dissolve.

Boiled another portion with HCl, it did not dissolve: added HNO_3 and heated again, the substance was not dissolved. Hence the substance is insoluble in water and in acids, and since it is white, it can only be $BaSO_4$, $SrSO_4$, ($CaSO_4$), $PbSO_4$, ($PbCl_2$), AgCl, SiO_2, Al_2O_3, SnO_2, Sb_2O_4, or CaF_2.

Poured Am_2S upon some of the substance: its colour was unchanged, *probable absence of Pb, Ag, Sn, Sb.*

Preliminary Examination.

Experiment	Observation.	Inference.
1. Took some of the powder up with a loop of moist platinum wire, ignited it strongly in the inner blowpipe flame, moistened it with HCl, and held in the Bunsen flame.	A yellowish green coloration.	Presence of Ba.
2. Fused a portion with Na_2CO_3 on charcoal in the inner blowpipe flame, placed the cooled mass on a bright shilling, and moistened it with a drop of water.	A black stain on the coin.	Presence of S.

Examination of the solution.

Fused some of the powder with fusion mixture on platinum foil: boiled the cold mass with water, filtered.

Filtrate: made acid with HCl and added $BaCl_2$, a white precipitate, which did not disappear on boiling the liquid:—*Presence of SO_4.*

The residue on the filter was well washed with boiling water, and boiling dilute HCl poured upon it, it dissolved completely:—

<table>
<tr><td rowspan="4">Absence of Group I.</td><td colspan="3">To a portion added strong H_2S-water and heated:</td></tr>
<tr><td rowspan="3"><i>No precipitate.</i>
Absence of Group II.</td><td colspan="2">To another portion added AmCl, then AmHO in excess, then Am_2S, and boiled:</td></tr>
<tr><td rowspan="2"><i>No precipitate.</i>
Absence of Group III.</td><td>To another portion of the original solution added AmCl, AmHO, and Am_2CO_3:</td></tr>
<tr><td><i>A white precipitate.</i> Presence of Ba, Sr, or Ca.
A platinum wire loop dipped into the original solution gave a yellowish-green tint to the Bunsen flame:
<i>Presence of Ba.</i></td></tr>
</table>

Found Ba, SO_4.

SECTION VI.

FULL ANALYTICAL COURSE AND TABLES.

Introductory Remarks —After the reactions in the Fourth Section have been tried, and the student has learnt how to detect the members of each Group when they occur singly or together, substances which may contain any members of the Groups should be analysed. The substance which is given for analysis by the following Tables will be either a liquid or a solid. Some hints, which will suggest the method of analysing a gaseous mixture, will be found in paragraph 73.

PRELIMINARY EXAMINATION OF A COMPLEX SUBSTANCE.

975. Before the student proceeds to apply the whole of the Systematic Course to the analysis of substances of complex composition, he will do well to examine a few substances for Metals and for Acid-radicles by the Preliminary tests only, which are given in paragraphs 985–1000. For a detailed account of any of the tests in these Tables the student must refer back to the description which is given of it in Section IV.

A little study of the Tables (989–1000) will show that they contain certain principal tests marked thus—"Exp. I." These stand in the first column. In the second column are placed the results which may be noticed on trying these experiments; a glance through these will forewarn the student of what has to be looked for.

The main classification of the observations which may be made is indicated by black-type letters; the subsidiary divisions are marked by small numbers and italics; and the individual results are simply placed in succession in a vertical column.

Occasionally an observation is made which it is advisable to confirm by an additional experiment. This Confirmatory test is entered in the first column just below the original observation, and is marked *Confirmatory* in order to distinguish it from the main experiment; its results are placed beside it in the second column.

The student should understand that any of the results, which are entered in the second column, may be obtained.

A few salts and mixtures, such as those mentioned in paragraph **1170**, should be subjected to a careful Preliminary Examination by the student. The results should be entered, *as soon as they have been obtained*, in the tabular form shown in paragraph **1107**.

FULL EXAMINATION OF SUBSTANCES IN SOLUTION.

976 After the student has become familiar with the Preliminary Examination of solid substances, he should fully analyse some easy substances which contain only a few metals and acid-radicles. A beginning is made at paragraph **987**, and after the Preliminary Examination has been finished, the substance is dissolved, and a Systematic Examination of its solution is made. The following paragraphs (**977-982**) give a general explanation of the method of examining the solution for metals, and of some points which require attention during the procedure which is stated in the General Table.

977. It is necessary first to separate the Metals into Groups, by adding the Group-Reagents in such order that each precipitates the metals of one Group only, and leaves the metals of all other Groups, which may be present, in solution.

This is effected by adding the Group-Reagents in the order which is directed in the General Table (**1007**). The

Group Table in paragraph 931 furnishes a convenient explanation of the scheme of the General Table.

The first Reagent added is HCl, which precipitates only the metals of Group I., and leaves the metals of Groups II., III., IV. and V. in solution. Hence if a precipitate is obtained when HCl is added in excess, it is filtered off; any member or members of Group I. will then be present upon the filter, while the members of all other Groups will remain in the filtrate.

When H_2S is passed into the Filtrate * metals of Group II. alone will be precipitated and may be filtered off.

*AmCl and excess of AmOH are added to the Filtrate,** after the H_2S has been removed from it, and Fe has been converted into ferric salt by boiling it with HNO_3. The AmHO will precipitate any members of Group III.A., while Group III.B. and Mg will remain dissolved by the AmCl, and Groups IV. and V. will also remain in solution.

*The addition of Am_2S to the Filtrate** will then separate Group III.B. as sulphides.

Group IV is then precipitated from the Filtrate * by the last Group-Reagent, Am_2CO_3.

The Filtrate * *is examined for Group V.*, the members of which are not precipitated by any Group-Reagent, and will therefore now remain in solution, if they were present in the original liquid.

978. Addition of Reagents in Excess.—Each Metal Group must be *completely* precipitated before the next Group-Reagent is added. If any precipitate has been formed by a Group-Reagent, it is accordingly necessary to add that reagent *in excess* before proceeding to add the next one. Unless the presence of each reagent in excess is made certain before proceeding, much confusion may be caused.

The general method for ascertaining that a Group-Reagent is present in excess consists in adding a few drops more of it to the clear liquid; the liquid must be rendered clear for this purpose either by filtration or by letting the precipitate settle. If any further precipitate is now formed, the whole of the liquid should be well stirred with more of the reagent, and the clear filtrate must then be again tested in the same way. This process must be repeated until no further precipitate is caused.

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* Or into the solution, supposing no precipitate to have been obtained, and filtration therefore to have been unnecessary.

In certain cases this method of proceeding is rendered unnecessary, since the presence of an excess of the reagent is detectable by its smell, after the solution and the reagent have been well mixed, and the air above the liquid has been blown out. This is the case with H_2S and with AmHO. The presence of an excess of a coloured reagent may usually be seen by the colour of the filtrate, as in the case of yellow Am_2S. If, however, an acid or an alkaline substance is directed to be added in excess, the student should always prove the presence of an excess by the use of test-papers.

979. **Washing Group Precipitates.**—Before a Group Precipitate is examined, it is necessary that it should be washed until it is free from adhering solution (96). This must also be done in the separations by filtration which are described in the Group Tables. If the washing has not been satisfactorily carried out, many complications may arise.

980. **Evaporation before Precipitating Group III.**—Certain organic substances hinder or prevent the precipitation of the metals of Group III.A. by AmHO. These substances are usually detected in the Preliminary Examination. If they have been detected, it is necessary to destroy them by evaporation and ignition before proceeding to precipitate Group III.

Evaporation to dryness and gentle ignition of the residue are also necessary in order to separate SiO_2, since, if this remained in solution, it might be mistaken for $Al_2H_6O_6$. The process also decomposes fluorides and removes HF.

But if organic substances, borates, fluorides, and silicates are known to be absent, evaporation is unnecessary, and it is sufficient to boil the filtrate from Group II. until it has no smell of H_2S; a few drops of strong HNO_3 are then added, and the boiling is continued for several minutes, in order to completely remove H_2S and to convert ferrous salts into ferric salts.

981. The Separation of the Sub-groups III.A. and III.B. from one another, by the method which is given in the General Table (**1007**), is never complete. For an account of more exact methods, the student is referred to paragraphs **1058**, *et seq.*

The method of examining the precipitate in Group III. may also be influenced by the presence of a phosphate in the solution: the change of procedure, and the causes which give rise to it, are explained in paragraph **1060.**

982. General Remarks.—It will be seen that the full course of analysis is so arranged that it serves not only to detect what is present in a substance, but also to prove that all else is absent. This is usually the object in view when the analysis of an unknown substance is undertaken.

The process of analysis may in some cases be shortened by using separate portions of the solution for each Group and test. The plan which is recommended in the General Table, of working throughout with the whole of the solution, is however preferable; since it enables the analyst to form an opinion as to the relative quantities of the different substances which are present, and this is usually important.

It will be unnecessary to do more than mention the extreme importance of the spectroscope for rapidly discovering certain substances, especially when they are present in minute quantity only (**113**).

An Example of Entry of Results is given in paragraph **1107**; it will be seen that the form adopted is that of the Analytical Tables.

Substances for Analysis are mentioned in paragraphs **1170**, **1171**, and **1172**.

The student will do well at first to gain experience in the analysis of easy mixtures, which are completely soluble in HCl and contain no cyanogen, organic salts, silicates, or phosphates precipitable in the Third Group. The analysis of such substances will be simple, since it will be unnecessary to evaporate the filtrate from the Second Group to dryness, or to follow any special method of precipitating and examining Group III.

When the ordinary course of analysis has been mastered, the analysis of substances, in which the above complications exist, should be attempted. and finally minerals, water residues, and artificial products and by-products should be analysed, in which minute traces of their constituents should be carefully looked for.

GENERAL ANALYTICAL TABLES.

GENERAL COURSE OF ANALYSIS FOR SOLID AND LIQUID SUBSTANCES.

983. **Cyanogen Test.**—Unless cyanogen is known to be absent, it must be tested for in a small portion of the substance by the tests described in paragraphs 648 and 650. If cyanogen is detected, refer to paragraphs 1083 *et seq.*, for a description of the preparation of the solution and of the method of analysis. If cyanogen is not present the substance is examined by paragraphs 985 and 986 if it is a liquid, and by paragraphs 987 *et seq.*, if it is a solid.

984. **Silicate Test.**—Silicate may be tested for by fusing some of the finely-powdered substance in a bead of microcosmic salt (600). If a liquid is being examined, a portion of it is evaporated to dryness, and the residue is fused into the bead.

I. THE SUBSTANCE FOR ANALYSIS IS A LIQUID.

985. **Reaction to Test-paper.**—Test the liquid with blue litmus-paper, and with turmeric-paper: one of the following results will be obtained (102).—

1. **It is Neutral,** not changing the colour of either paper, this shows the absence of acids and alkalis and of salts with acid or alkaline reaction. The salts of Ag and Mg and certain salts of Am, Na, K, Ba, Sr, and Ca are the only neutral soluble salts.

2. **It is Acid**, turning *blue* litmus *red*. this proves the presence of an acid, or of a salt with acid reaction.

3. **It is Alkaline**, turning turmeric *brown*: indicating the presence of Am, Na, K, Ba, Sr, or Ca as hydroxide, or of a salt with alkaline reaction.

986. **Test for Dissolved Solid.**—Evaporate a few drops of the liquid upon platinum foil, thin glass, or porcelain, smelling it occasionally:—

1. **A Residue is left.**—Examine the liquid by the further preliminary tests in paragraph 937. or evaporate a portion of the solution to dryness in a porcelain dish, avoiding heating the substance after it is dry, and examine the residue by the Preliminary Tables for Metals and Acid-radicles (987–1000). Examine the larger part of the solution for Metals by the General Table (1007), paying attention to paragraph 1008: reserve the rest of the solution for the Examination for Acid-radicles by paragraphs 1042 *et seq.*

2. **No Residue is left.**—The liquid must consist of some volatile substance, probably of water. This is either pure, or it contains certain gases or volatile substances, such as $(NH_4)_2CO_3$, CO_2, NH_3, HCl, Br. These may be detected by their smell, by the action of the liquid on litmus paper, or by special tests (73). If the liquid has no action on litmus and no smell, and if it leaves no residue on evaporation and is tasteless, it is pure water.

II. THE SUBSTANCE FOR ANALYSIS IS A SOLID.

987. Examine the General Properties of the Substance.

If the substance is **lustrous and metallic**, refer to paragraph 1067 for the method of analysis; if it is non-metallic in appearance, proceed to examine it as follows.

Note down anything that can be learned about the physical properties of the substance by the use of the senses aided by a lens or microscope, and by a magnetised penknife-blade.

Note especially whether the substance consists of more or less sparkling particles with similar geometrical shape, when it is said to be **crystalline**; or whether the particles are irregular in shape, and **amorphous**.

Also test its **hardness**, by ascertaining whether the substance is readily powdered.

If the particles are sufficiently large, and especially if they are crystalline minerals (1172), ascertain the degree of hardness more carefully, since this will be of use in identifying the substance. Very soft minerals can be scratched and marked by the thumb-nail (Talc, Steatite). A harder substance which is not affected by the thumb-nail may be scratched by a bronze coin (Cryolite, Cinnabar). Harder minerals still will only be scratched by a penknife (Apatite, Fluorspar). Very hard substances, such as quartz, cannot be scratched by a knife, but the knife produces a steel stain upon their surface.

Try whether the substance is **magnetic** by immersing the tip of a magnetised blade into the fine powder, and seeing whether its particles are attracted. Fe and certain of its compounds are the most powerfully magnetic bodies known, but Mn, Ni, Co and certain of their compounds are also magnetic.

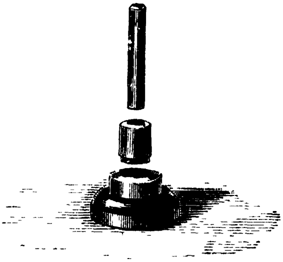
Note also whether the substance possesses any characteristic **smell** or **colour**. The following are some of the more commonly occurring coloured compounds:—

Blue: hydrated cupric salts, and anhydrous cobalt salts.
Green: certain Fe″-salts are pale green; $CuCl_2$, salts of Ni, manganates, and certain compounds of Cr, are intense green.
Yellow: HgO, As_2S_3, CdS, SnS_2, chromates, Fe‴-salts.
Red: HgO, HgI_2, HgS, Pb_3O_4; dichromates are orange-red.
Pink. salts of Mn are delicate pink; hydrated salts of Co are reddish-pink.
Brown: Fe_2O_3, PbO_2; PbO and CdO are light brown.
Black: CuO, MnO_2, Sb_2S_3, FeS.
White: anhydrous salts of Cu and Fe″, and many other substances
Colourless: a large number of colourless substances are known; these, as well as many of the faintly coloured bodies, yield *white* powders when they are crushed.

988. Reduction to fine powder.—After the above examination has been completed, the solid substance is reduced to the finest powder possible, by rubbing it in the mortar. An ordinary Wedgwood mortar may be used for powdering substances which are pretty soft; but hard rocks and minerals frequently require to be first crushed in a clean bright steel mortar (fig. 79) by blows of a hammer, and to be afterwards pulverised in an agate mortar.

It must be understood that the subsequent chemical examination is very greatly facilitated if the substance has been carefully reduced to an impalpable powder, which does not feel gritty when it is rubbed beneath the pestle or between the fingers.

FIG. 79.



STEEL MORTAR.

A portion of this powder is submitted to the tests which are described in the following Preliminary Examinations for Metals and for Acid-radicles: and the remainder is reserved for the fuller method of examination in solution (**1001** *et seq.*).

Experiment.	Observation.	Inference.
989. **Exp. I.—Heat a small quantity of the substance in a small test-tube or ignition-tube.**	**A. The substance does not change.**	Absence of *organic* substances which blacken and give off a smell of burning; of *volatile* substances which sublime, of substances which are altered by ignition; and of *water* combined with or absorbed by the substance, which would be evolved as steam and form drops on the side of the tube.
	B. The substance changes. 1 *It changes colour*:—	
	Yellow, hot | *white,* cold	ZnO.
	Yellow, brown „ | *yellow* „	PbO.
	Yellow-brown „ | *yellow* „	SnO_2 or Bi_2O_3.
	Dark red or *black* „ | *red-brown* „	Fe_2O_3.
	The substance blackens— *a* Emitting a smell of burning. The black carbon may be burnt off by strongly heating on platinum foil with the blowpipe flame.	Presence of organic matter.
	Smell of acetone.	An acetate.
	Smell of burning sugar	A tartrate.
	b Emitting no smell of burning The colour is not removed by ignition as under *a*.	Probable presence of Co- or Cu-salts.
	2 *It fuses, and becomes solid again on cooling.*	Salts of alkalis or certain salts of the alkaline earths
	3. *It sublimes.*	Compounds of Am, As, Hg; or free S or I.
Confirmatory.—The sublimate is carefully examined, if necessary, with the aid of a lens.	White, crystalline (sparkling) sublimate.	$HgCl_2$, As_2O_3.
	White, non-crystalline sublimate	Probably Am-salt.
	Black sublimate, becomes red when rubbed.	HgS
	Yellow sublimate.	As_2S_3 HgI_2, becomes scarlet when rubbed.
	Sublimate of reddish drops, which are yellow when cold.	S free or from certain polysulphides.
	Violet vapour, cooling to blackish crystals.	Presence of I.
Confirmatory.—Heat another portion of the substance, mixed with about three times as much Na_2CO_3 in fine dry powder and a little KCy, in an ignition-tube (282).	Grey mirror, which, when rubbed with a splinter of wood or a glass rod, or when examined by a lens, is seen to consist of globules of Hg.	Presence of Hg.
	Blackish-brown shining mirror, and smell of garlic; no globules.	Presence of As.

Experiment.	Observation.	Inference.
Confirmatory —Pour a little strong KHO solution upon some of the substance and heat to boiling; or mix some of the substance with soda-lime in a mortar, and moisten and heat.	NH_3 is given off, known by its smell and by turning moist red litmus-paper blue or turmeric-paper brown; the change in colour is only a trustworthy indication if it occurs very soon, as the NH_3 in laboratory air may cause it to occur after a time.	Presence of NH_4.
	4. *The substance gives off water.*	Presence of water, absorbed or combined.
	It fuses first, then gives off water, and again becomes solid if the heat is continued.	Water of crystallisation.
	It swells up considerably whilst giving off its water	Borax and certain borates, alums, and phosphates
Confirmatory —Examine the drops of water on the sides of the tube with blue and red litmus-papers.	The water is alkaline.	Probably NH_4-compounds.
	The water is acid.	Presence of volatile acids, *e g.*, HNO_3, HCl, H_2SO_4.
	5 *Gas or vapour is given off**. *a It is without smell*	
Confirmatory. — Introduce a burning splinter of wood into the tube.	It burns more brightly, and if introduced with a spark at the end is inflamed,	O from chlorates, nitrates, peroxides, &c., or possibly N_2O.
	The flame is extinguished	CO_2 or N.
Confirmatory — Introduce a glass rod, moistened with lime-water, into the tube.	The lime-water turns milky.	CO_2 from carbonates, oxalates, &c
	The lime-water does not turn milky, but the flame is extinguished.	N from $AmNO_2$ probably.
	b. The gas or vapour has a smell.	
Confirmatory — Introduce a glass rod or slip of paper moistened with $K_2Cr_2O_7$.	Smell of burning S The $K_2Cr_2O_7$ turns green; moist blue litmus is also reddened by the gas	SO_2 from combustion of free S, or from acid sulphites, thiosulphates, reduction of sulphates or oxidation of sulphides
	Reddish-brown nitrous fumes are given off, known by their peculiar smell, and by not colouring starch paste orange red.	Nitrates of heavy metals (*e g.* of Pb, Bi, Hg. &c.)
	Smell resembling Cl:—	
Confirmatory. — Introduce a glass rod carrying a piece of moist litmus.	Yellowish gas, which bleaches moist litmus.	Cl from certain chlorides.
Confirmatory. — Introduce a glass rod with moist starch powder on its end.	Brown vapour, which colours the moist starch powder orange red.	Br from certain bromides.
	Violet vapour, which colours starch solution blue.	I, free or from certain iodides.
	NH_3 is smelt.	NH_4 or cyanogen compounds.

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* This will usually be shown by effervescence of the substance; if the gas or vapour is not detected by its colour or smell, it can only be found on testing for it specially by the confirmatory tests below.

Experiment.	Observation.	Inference.
	c The gas can be ignited, and burns at the mouth of the tube with a —	
	Pale *yellowish green* flame, more or less explosive.	NH_3 probably from strongly heated NH_4NO_3.
	Bright *white* flame, producing white fumes; the unburnt gas smells of garlic.	PH_3 probably from a hypophosphite.
	Peach-blossom coloured flame.	Cy from a cyanide, probably from $HgCy_2$.
990. Exp. II.—Dip a moistened loop of platinum wire, which gives no colour to the Bunsen flame, into the substance, and hold the loop with the adhering powder near the top of the Bunsen flame, or in the inner blowpipe flame. Then moisten with a drop of strong HCl and heat again. Continue the heating until no further change occurs in the colour of the flame.	A The substance colours the flame:—	
	1 *Intense yellow*	Na.
	Examine the flame through the indigo-prism, a crimson colour is seen.	Presence of K or Sr, or both.
	2 *Pale lilac*, crimson through the indigo-prism.	K.
	3 *Yellowish green.*	Ba probably.
	4 *Crimson*, same through the indigo-prism.	Sr.
The flame coloration should also be carefully examined by means of the spectroscope (114, 115).	5. *Orange red*, dingy green through the indigo-prism.	Ca.
	6. *Bright green*	Cu or B_2O_3.
Note If silicic acid is present, the colorations for K and Na usually show only after ignition of the powdered substance with powdered $CaSO_4$.	A blue colour is seen after moistening with strong HCl.	Cu.
	7 *Blue* { *Intense.*	$CuCl_2$, $CuBr_2$
	Livid.	As, Sb, Pb, $HgCl_2$, $ZnCl_2$, $SnCl_2$.
	B The flame is not coloured.	Above metals absent.
991. Exp. III.—Heat the finely powdered substance in a small cavity scooped in a piece of wood-charcoal, in the blowpipe flame.	A The substance decrepitates or crackles.	NaCl and certain other crystalline salts.
	B The substance deflagrates, or causes the charcoal to burn rapidly.	Chlorates, nitrates, &c.
	C. The substance fuses easily, and is absorbed by the charcoal, or forms a liquid bead.	Salts of alkalis and certain salts of the alkaline earths.
	D. An infusible residue is left on the charcoal:—	
	1. *The residue is white and very luminous.*	Probably BaO, SrO, CaO, MgO, Al_2O_3, ZnO (or SiO_2)
Confirmatory.—Detach a portion of the cool residue from the charcoal, place it upon a piece of red litmus-paper, and moisten with a drop of water.	The paper turns blue, showing the residue to be alkaline.	BaO, SrO, CaO, and possibly MgO.

Experiment.	Observation.		Inference.
Confirmatory.—Moisten the residue on the charcoal when cool with several drops of $Co(NO_3)_2$ solution, and heat again strongly in the outer blowpipe flame.	A *blue* residue, the colour of which does not disappear with intense heat		Al_2O_3, and some phosphates, arsenates, silicates, and borates.
	A *pink* residue.		MgO.
	A *green* residue.		ZnO (or possibly Sn).
Confirmatory.—Heat a small quantity of the substance in a clear colourless borax bead, first in the outer, then in the inner blowpipe flame.	2. *The residue is coloured.* Colour of bead:		
	In outer flame.	In inner flame	
	Green, hot, and *blue*, cold	*Colourless*, or *red*, cold.	Cu.
	Blue, hot and cold	*Blue*, hot and cold.	Co.
	Violet, hot, and *yellow*, cold	*Grey* or *opaque*.	Ni.
	Brown-red, hot, *yellow*, cold	*Olive green*, hot and cold	Fe.
	Green, hot and cold.	*Green*, hot and cold.	Cr. See *Confirmy* test below.
	Reddish purple, hot and cold	*Colourless*, hot and cold	Mn.
Confirmatory.—The presence of Mn and Cr, may be confirmed by fusing the substance with Na_2CO_3 and KNO_3 on platinum foil.	*Light-yellow* mass on cooling		Cr.
	Blue-green mass on cooling.		Mn (See **993**)
Confirmatory.—Heat also some of the substance, mixed or covered with powdered KCy and Na_2CO_3 or $K_2C_2O_4$, on charcoal in the inner blowpipe flame.	3 *The residue is coloured, or metallic scales or globules are seen* The substance is reduced to the metallic state (See **994**)		Au, Ag, Cu, Pb, Sn, Sb, Bi; [Co, Ni, Fe, Mn, form grey powders].
	a. Without incrustation		Au, Ag, Cu, Co, Ni, Fe, Mn, Sn.
	Brilliant white metal.		Ag, Sn: Ni if hard.
	Yellow metal		Au.
	Red scales or globules		Cu.
	Grey powder attracted by a magnetised knife-blade (994)		Fe, Co, Ni, Mn.
	b With incrustation.		
Confirmatory.—Detach several of the globules with the point of a knife, and strike them on the bottom of an inverted mortar with the pestle; if they flatten to a cake they are *malleable*, if crushed to a powder they are *brittle* Try also if a globule, fixed on the point of a knife, marks paper.	Globules.	Incrustation	
	White, malleable, not marking paper	None, or very slight.	Sn.
	White, brittle.	*Orange*, hot. *Yellow*, cold.	Bi.
	White, malleable, marking paper	*Yellow*, hot and cold.	Pb.
	White, brittle, giving white fumes.	*White*, close to substance.	Sb.
Note.—The appearance and malleability of one reduced metal is frequently much altered by the presence of another	*None*	*Yellow*, hot. *White*, cold.	Zn.
	None	*Red-brown*, easily volatilised.	Cd.
	None.	*White*, and white fumes; smell of garlic.	As.

Experiment.	Observation.	Inference.
992. Exp. IV.—Fuse some of the substance, mixed with Na_2CO_3, on charcoal in the inner blowpipe flame produced from a spirit-lamp; remove the fused mass when cold, place it on a bright silver coin, moisten with a drop of water, and let stand for several minutes.	On rinsing off the substance, the coin is found to be stained black (501). Also, if a drop of HCl be placed upon the mass, a smell of H_2S is perceived, and lead-paper is blackened.	Presence of S, free or combined. *Note.* — Since this reaction serves to detect S and sulphur-acid-radicles, it more properly belongs to the Preliminary Examination for acid-radicles it is, however, best tried here.

NOTES TO THE PRECEDING PRELIMINARY TABLE.

993. The green colour produced by Mn conceals the light yellow due to Cr; on boiling the residue with water it gives a pink or purple solution, the colour of which is best seen on filtering: this colour is due to the formation of $KMnO_4$, and shows Mn to be present. On acidifying this solution with $H\bar{A}$ and boiling again for several minutes, the purple colour is destroyed; and on filtering, the yellow colour due to Cr is seen: the presence of Cr may be further confirmed by the formation of a yellow precipitate in the acid solution on addition of $Pb\bar{A}_2$.

994. The metal is best separated and examined by detaching the mass when cold from the charcoal, and powdering it by crushing in a mortar or on a watch glass with a little water, letting stand for a short time, and then quickly pouring off the water down a glass rod or pestle which is wetted and pressed against its edge; by several times repeating this operation, the heavier metallic particles alone are left in the mortar or watch glass (111).

995. *Note.*—When the substance to be examined contains several bodies, they frequently more or less mask one another's reactions: thus Co if mixed with Fe will give a bead green whilst hot and blue when cold, thus resembling Cu, but differing in remaining blue in the inner flame: hence the composition of many complex mixtures is only roughly indicated by the Preliminary Examination, and must be confirmed and established in the wet way. Many substances, more particularly minerals, however, can be completely analysed by a careful Preliminary Examination.

PRELIMINARY EXAMINATION FOR ACID-RADICLES.

996. The substance is required for this examination in the state of powder or of *strong* solution.

If the substance given for analysis is a solution, part of it may be evaporated to dryness · the dry residue is then finely powdered and subjected to the following tests. If several acid-radicles and metals are present, the reactions may be more or less perfectly concealed or altered, hence failure in obtaining a certain reaction does not in all cases necessarily prove the absence of the corresponding acid-radicle.

Experiment.	Observation.	Inference.
997. Exp. I.—Treat some of the substance with dilute HCl and note the result, then heat moderately.	*One or more of the following gases may be evolved* —	
	A colourless gas without smell, which turns milky a drop of lime-water on the end of a glass-rod	CO_2 from a carbonate.
	A gas of suffocating smell, which turns a drop of $K_2Cr_2O_7$ solution green.	SO_2 from a sulphite, or from a sulphate in the presence of As_2O_3 or other reducing substance
	Yellow S is precipitated at the same time	SO_2 and S from a thiosulphate.
	A gas with fetid odour, which blackens a drop of $Pb\bar{A}_2$ solution, or a piece of lead-paper. (Refer to 892)	H_2S from a sulphide; or possibly from a sulphite or thiosulphate, if Zn or other reducing agent be present.
	Reddish fumes.	From a nitrite.
	A yellowish-green gas, of suffocating smell, which bleaches moist litmus-paper, is evolved :—	
	a. In the cold; and also evolved on adding $H\bar{A}$.	Cl from hypochlorite
	b. Only when heated.	Cl from action of HCl on oxidising substances, *e.g.*, MnO_2, chromates, nitrates, chlorates, &c.
	A colourless gas, smelling of bitter almonds.	HCy from a cyanide.
Dip into the HCl solution a strip of turmeric-paper.	On drying this paper at a gentle heat, it becomes reddish-brown.	Presence of a borate (588).

Experiment.	Observation.	Inference.
998. Exp II. — Heat another portion gently with strong H_2SO_4 No attention need be paid to gases evolved in Exp. I (997)	1. A gas is evolved with pungent smell, which fumes in the air, and renders milky a drop of $AgNO_3$ solution made acid with HNO_3	HCl, HBr, HI, HF* from chloride, bromide, iodide, or fluoride
Confirmatory — Mix some of the substance with MnO_2 *free from chloride*, add strong H_2SO_4 and warm.	A *yellow* gas is given off with strong smell, which bleaches moist litmus-paper (1000)	Probably Cl from chloride
	Brown vapour is given off, which colours a little moist starch powder orange-red.	Br from bromide
	Violet vapour is given off, which colours a drop of starch paste blue	I from iodide
	2 A heavy fuming suffocating gas is evolved, and the liquid behaves as if the glass were greasy on rinsing out the tube, and drying it thoroughly, the inside is seen to be corroded and dimmed	HF from fluoride: confirm by 606 The HF acting upon the silica of the glass evolves SiF_4, which is detected by holding a moistened glass rod in the gas, SiO_2 will be deposited as a gelatinous film upon it
Confirmatory —Drop into the hot liquid a few small pieces of copper If no brown fumes are seen proceed to next test	3 The acid fumes are reddish, usually seen only on *heating* after adding a few fragments of Cu.	Presence of nitrate If iodide is present it must usually be separated by $CuSO_4$ (563) before a nitrate can be detected.
Confirmatory — Boil some of the solid substance with water, or take some of the liquid, cool, add about twice as much strong H_2SO_4, cool, and pour cold solution of $FeSO_4$ carefully in upon the top of the acid liquid.	A dark brown ring or layer forms upon the surface of the acid, either at once or on cooling the liquid by immersing the test-tube in cold water. [Refer to *Note*, 1050.]	Presence of nitrate.
	4 The acid is coloured reddish-yellow, and a yellow gas is evolved which smells like Cl and bleaches litmus; on heating the acid explosion or crackling occurs. Confirm by 545.	Presence of chlorate For detection of nitrate and chlorate when mixed, see 550.
	5 The substance changes from yellow to green, O being evolved, as is shown by a glowing taper.	Presence of chromate.

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* HF would act upon the glass, and the SiF_4 thus formed would render the drop milky by depositing SiO_2: distinguished by corroding the glass. (See 998, 2.)

Experiment.	Observation	Inference.
	6 Yellowish green gas, with suffocating smell, which bleaches moist litmus	Cl from a hypochlorite (detected already in 997), or from a chloride in presence of MnO_2 &c.
	7. The same gas as in 6, but coloured *reddish brown* by nitrous fumes. or coloured reddish brown by $CrOCl_2$.	A chloride in presence of a nitrate or nitrite. A chloride in presence of a chromate.
	8. Substance does not blacken, but evolves CO which burns with a blue flame, and CO_2 which turns a drop of lime-water milky.	Presence of an oxalate.
	9. Substance blackens, and SO_2 is smelt.	Presence of a tartrate.
	10 A smell of acetic acid or vinegar is noticed.	$H\bar{A}$ from an acetate.
Confirmatory.—Add a little alcohol to the substance and heat with H_2SO_4.	Strong fragrant odour.	Ethyl acetate from an acetate.

NOTES ON THE PRECEDING TABLE.

999. The S present in sulphides which are not decomposed by dilute HCl, is detected by fusing the powdered substance with two or three times as much fusion mixture ($Na_2CO_3 + K_2CO_3$) in a covered crucible, boiling with a little water, and placing a drop upon a bright silver coin, when a dark stain will be produced; or by adding excess of an acid to the solution, when H_2S will be evolved. Smaller quantities of S may be detected by the formation of a black colour or precipitate on adding $Pb\bar{A}_2$ to the solution of the fused mass.

1000. Chlorides treated in this manner evolve Cl, recognised by its colour, smell, and bleaching action on litmus-paper; but this test for chlorides is trustworthy only if the precautions stated in 554 are attended to, since many samples of MnO_2 evolve Cl when they are warmed with H_2SO_4.

EXAMINATION FOR METALS IN THE WET WAY.

After the Preliminary Examination of the solid substance has been completed, a portion of the substance is dissolved (**1001**), and the solution is submitted to the systematic examination, which commences at paragraph **1007.** This is often termed "The Examination in the Wet Way."

Process of Solution.

1001. A portion of the solid substance, reduced to an impalpable powder (**988**), is boiled in a flask (fig. 38, p. 61) or boiling-tube with distilled water. if it dissolves entirely, proceed to **1007.**

If the powder does not dissolve, allow the liquid to stand until the undissolved portion has in great part settled, then decant through a filter: the filtrate is Solution I.:—

<table>
<tr><td colspan="4"><i>Residue in flask</i> boil with dilute HCl (1004), allow any residue to settle, decant through the filter used above (1005); repeat this process and finally boil the residue with strong HCl, let settle, decant through the filter:—</td></tr>
<tr><td rowspan="3"><i>Filtrate is</i>
Solution II.</td><td colspan="3"><i>Residue:</i> heat with dilute, then with strong HNO_3 (1006) as was directed above for HCl; decant through the filter.—</td></tr>
<tr><td rowspan="2"><i>Filtrate is</i>
Solution III.</td><td colspan="2"><i>Residue</i> may be warmed for a short time with a mixture of strong HNO_3 (1006) with three times as much HCl; if this does not dissolve it, dilute and decant the acid mixture through the filter:—</td></tr>
<tr><td><i>Filtrate is</i>
Solution IV.</td><td><i>Residue:</i> wash well with water, dry, and examine as directed (1074 <i>et seq.</i>) for substances insoluble in water and acids.</td></tr>
</table>

1002. Solutions I., II., III., IV. need not be examined separately. Add to I. a few drops of dilute HNO_3, and if no precipitate is produced (**1008**) acidify it with HCl; add also to Solution III. some HCl. Any precipitate caused by HCl in these solutions, is filtered off and examined by Table I. (**1022**). The Solutions I., II., III., IV. are then mixed together, and any precipitate produced by mixing them, is examined by paragraphs **1074** *et seq.* as an insoluble substance.

It is best to boil down Solutions III. and IV. considerably, in order to get rid of most of the HNO_3 which they contain, before they are mixed with Solutions I. and II.

The mixed Solutions are then examined by paragraph **1007.**

1003. If a complex solid mixture is given for analysis, and the only object is to obtain a solution as rapidly as possible, the substance may be at once boiled with aqua regia for a few minutes, then diluted and filtered The residue is examined as a substance insoluble in water and acids (1074, *et seq*). The filtrate is evaporated nearly to dryness, dilute HCl is added, and the solution is examined by the General Table (1007).

1004. Any Changes which occur on the Addition of HCl should be noted down. Notice especially whether any gases are given off. Many of these may be at once recognised by their smell; but a closer examination may be neglected, as these gases will have been already obtained in the Preliminary Examination for Acid-radicles (**997**).

If the gases are evolved from the aqueous solution when HCl is added, or when the HCl and aqueous solutions are mixed, the corresponding acid-radicles must have been present in salts soluble in water, probably salts of alkali-metals. If the gases are evolved when the residue insoluble in water is treated with HCl, the radicles were present in salts insoluble in water.

1005. When the hot HCl-solution is cooled, crystals often separate. These usually consist of $PbCl_2$, rarely of $BaCl_2$. The crystals should be filtered off and dissolved in a little boiling water. Ba is easily found by the flame-coloration which is given by the solution (**175**). The formation of a *bright yellow* precipitate, when a drop of K_2CrO_4 is added to the solution, proves the *Presence of Pb*. Ba gives only a *pale yellow* precipitate.

If Pb has been found, and a residue is left after the original substance has been treated with HCl, this residue may consist of $PbCl_2$: this substance may be dissolved by boiling it with water, and the use of HNO_3 may be thus avoided (**1006**).

1006. HNO_3 seldom requires to be used in dissolving a substance. This acid should be employed, when necessary, only in very small quantity, since it decomposes H_2S with separation of S unless the solution is dilute and cold (**1011**): the presence of HNO_3 may accordingly delay or prevent the precipitation of Group II.

1007. GENERAL TABLE FOR SEPARATION OF

These lines run across both pages.

If the substance for analysis is a liquid with *neutral* or *alkaline* reaction, it examined as directed below.

In this Table, and also in the Group Tables which follow, it has been sup- precipitate will be produced when the reagent is added for its detection ; and

Add to the greater part of the "original solution"* HCl; if any precipitate is produced,		
The precipitate may contain— $PbCl_2$—*white.* $AgCl$—*white* Hg_2Cl_2—*white* (See 1010) **Examine the precipitate by Table I. (1022)**	The filtrate or solution, which must give no further precipi- then diluted with water (1013), unless it is already dilute; several minutes (1014, 1015) and filtered ·	
	The precipitate may contain— HgS—*black* PbS—*black* Bi_2S_3—*black* CuS—*black* CdS—*yellow* Insoluble in KHO and in Am_2S. SnS—*brown* SnS_2—*yellow* Sb_2S_3—*orange* As_2S_3—*yellow* [Au_2S_3—*black*] [PtS_2—*black*] Soluble in KHO and in Am_2S. **Examine the precipitate *at once* by Table II. (1024)**	The solution or filtrate is di- cipitated as soon as H_2S dish until it ceases to smell and the residue gently ignited heated, then water is added; Add a few drops of the acid tate shows the *presence of a* Add to the rest of the HCl solu- any precipitate, which may (1018) if a phosphate is phates are absent proceed as [*Note* —**Refer to**
Note.—When the hot HCl solution is cooled, $PbCl_2$ often separates in white crystals. The absence of Pb is not proved, if it is not precipitated in this group, as $PbCl_2$ is somewhat soluble, and Pb may therefore be found only in Group II.	*Note* 1.—Any precipitate formed on boiling the filtrate will have to be added to the above. *Note* 2 —Unless the liquid has been sufficiently diluted some of the above metals may be afterwards precipitated by Am_2S in Group III., and cause much confusion.	**The precipitate may contain—** $Al_2(HO)_6$—*almost colourless.* $Cr_2(HO)_6$—*light green* $Fe_2(HO)_6$—*reddish brown.* Examine the precipitate by Table III.A (1027).

NOTES ON THE GENERAL TABLE.

1008. If the liquid under examination is alkaline or neutral in reaction, HNO_3 must be added in slight excess, before the examination by the General Table is commenced ; a change of colour from green to purple indicates the *Presence of Manganate* (628, 629). If no precipitate forms, the liquid is at once examined by the General Table ; if a precipitate is produced, more HNO_3 is added, and the

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* By the "original solution" is meant the solution of the substance if a solid, or the substance itself if a liquid, to which no re-agent has been added.

METALS INTO GROUPS BY GROUP-REAGENTS.

requires special treatment (1008); if its reaction is *acid*, it may be at once

posed that all metals are present: if a metal or group of metals is absent, to
hence no filtration is necessary, the solution being then treated as a filtrate.

continue to add HCl as long as it causes any further precipitate, warm and filter:

tate on addition of several drops of HCl, is boiled down if necessary (1011), and is
it is then saturated with H_2S by passing the gas through the solution* (1124) for

luted and H_2S is passed again into it, to make sure that Group II is completely pre-
produces no further precipitate the clear solution or filtrate is boiled in a porcelain
of H_2S (1016), a little strong HNO_3 is then added, and the liquid is evaporated to dryness
in the dish When the dish is cool, a little strong HCl is poured upon the residue and
any undissolved residue is filtered off (1017)

solution to some $AmHMoO_4$ solution in a test-tube, and warm gently; a yellow precipi-
phosphate, if no precipitate is formed with $AmHMoO_4$, *phosphates are absent.*

tion AmCl, heat it to boiling, then add AmHO in excess, boil again, and filter off
form, as quickly as possible, keeping the funnel closely covered with a glass plate
present and a precipitate is produced by AmHO refer to Tables (1065, 1066) if phos-
is directed below —

1062, *et seq.*, for full directions for precipitating Groups III.A and III B]

Add to the filtrate or solution, which may be coloured (1019), yellow Am_2S in excess,
boil and filter:—

<table>
<tr><td rowspan="2">The precipitate may contain—
ZnS—<i>white</i>
MnS—<i>light-pink.</i>
CoS—<i>black</i>
NiS—<i>black.</i>
Examine this precipitate <i>at once</i> by Table III.B (1028)</td><td colspan="2">Add to the filtrate or solution, which must be distinctly yellow or brown (1020), Am_2CO_3, warm gently and filter.—</td></tr>
<tr><td>The precipitate (1021) may contain—
$BaCO_3$—<i>white.</i>
$SrCO_3$—<i>white.</i>
$CaCO_3$—<i>white.</i>
Examine the precipitate by Table IV. (1031)</td><td>The filtrate may contain Mg, K, Na.
Examine by Table V. (1035).</td></tr>
</table>

liquid is heated, and if a precipitate still remains, it is filtered off and the filtrate is examined by the General Table, commencing with the addition of HCl.

The precipitate caused and not dissolved by HNO_3 may be finely divided sulphur, which separates as a *white* powder from a sulphide, and as a *yellow* powder from a thiosulphate†; this precipitate may be recognised by its colour, and by not being separable by standing or filtration: it may be disregarded. There may also be precipitated

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* Instead of diluting and passing the gas, H_2S-solution may be added.

† Formerly called a "hyposulphite."

H_4SiO_4 (gelatinous, almost colourless), SnO_2 or metastannic acid (1009), Sb_2O_5, AgCl (all three white), SnS_2, As_2S_3 (both yellow), Sb_2S_3 (orange). A full examination of this precipitate for all that it may contain may be made by boiling the precipitate with aqua regia, and then diluting the liquid and filtering it ; any insoluble residue which remains is then examined as a substance insoluble in water and acids (1074) ; the aqua regia solution is added to the filtrate from the precipitate which was originally produced by HNO_3, and any precipitate which is caused by mixing these solutions is examined by Table I. (1022).

1009. Metastannic acid is precipitated from the solution of an alkaline metastannate by the addition of an acid. It forms a white precipitate, which becomes *golden-yellow* when it is moistened with $SnCl_2$-solution. It is further recognised by dissolving in the washing-water while it is being washed, and being reprecipitated when the washing runs into the acid filtrate.

1010. It must be remembered that strong HCl also precipitates a strong solution of a barium salt. This precipitate, however, is readily dissolved when water is added and the liquid is heated . it is easily distinguished in this way from AgCl and Hg_2Cl_2.

1011. If HNO_3 or aqua regia has been used in dissolving the substance (1001, 1006), or if the solution smells of Cl or SO_2, the liquid should be boiled down considerably, until neither of the above gases is smelt when HCl is added and the liquid is again boiled. The solution is then diluted, and H_2S is passed into it at once, whether the dilution has caused a precipitate or not (1013).

If As has been detected in the Preliminary Examination, H_2SO_3 should be added, until the liquid smells of SO_2 after it has been boiled for a short time. The liquid is then heated for some time short of boiling until it no longer smells of SO_2. This treatment reduces arsen*ic* and stann*ic* compounds to the arsen*ious* and stann*ous* condition.

In the case of As this reduction is to be recommended, since arsen*ic* compounds are not easily precipitated by H_2S. It is also very desirable to reduce stannic compounds, since SnS_2, unlike SnS, readily runs through the filter, and being yellow in colour may be mistaken for S. It will of course be necessary to try the special tests 323-326 and 340–342 upon the *original solution*, in order to ascertain in which state As and Sn were originally present. If any precipitate, other than white sulphur, is produced by boiling with H_2SO_3, refer to paragraph 1012.

1012. While the solution is being boiled with H_2SO_3, some H_2SO_4 is usually formed. This acid may partially or completely precipitate

Pb, Ba, Sr as white sulphates. H_2SO_3 may also precipitate Au as a fine powder, which causes the cool liquid to appear blue by transmitted and brown by reflected light, this fine precipitate remains suspended but separates as a black powder when the liquid is boiled. Examine any precipitate which has been formed by H_2SO_3 by Table G (1023)

1013. Dilution with water may cause Bi, Sb, Sn to form white precipitates of their oxychlorides. These precipitates may be disregarded, since H_2S readily converts them into sulphides.

1014. H_2S often produces a fine *white* precipitate of S : this is caused by the presence of certain oxidising substances, such as Cl, HNO_3, $HClO_3$, H_2CrO_4, $HMnO_4$, H_2SO_3, Fe_2Cl_6 (1011). This precipitate is known by its perfect whiteness, and by not being separable by standing or by filtration, it may be neglected. Great care must however be taken not to mistake SnS_2 for S ; it resembles S in running through filter-paper, but differs by being distinctly *yellow* in colour ; it may often be coagulated by shaking or heating the liquid.

1015. Certain changes may be observed while H_2S is being passed ; they should be carefully noted. Thus while Hg and Pb are being precipitated by H_2S, the precipitates often show characteristic colours (279, 284). A solution, which is coloured reddish-yellow by H_2CrO_4, becomes green ; a solution, which is coloured purple by $HMnO_4$, becomes colourless ; and a reddish-yellow ferric solution becomes a pale-green ferrous solution. Each of these changes is attended by separation of sulphur.

1016. If a yellow precipitate forms when the filtrate or solution has been saturated with H_2S and then boiled, this shows the presence of an arsen*ic* or a stann*ic* compound. In this case H_2S should be passed into the boiling liquid as long as it causes any further precipitate, the precipitation being preceded with advantage by reduction with SO_2 (1011). The precipitate is filtered off and added to any precipitate which has been already obtained by H_2S, and is then examined by Table II.

1017. SiO_2 and other substances may be present in the insoluble residue which is left after evaporation with HCl. SiO_2 is distinguished from any other substances by disappearing when it is heated with HF.

Al_2O_3, Fe_2O_3, Cr_2O_3 may remain undissolved by HCl, if they have been rendered difficultly soluble by the ignition to which the residue has been subjected. They may be dissolved by long-continued heating with strong HCl. The solution thus obtained is added to

$BaSO_4$ and $SrSO_4$ may also remain undissolved. They are detected by heating some of the residue strongly for a short time in the inner blowpipe-flame upon a loop of platinum wire, then moistening the loop with a drop of strong HCl, and holding it in the outer part of the Bunsen-flame. Crimson-red flashes, which appear red through the indigo prism, prove the presence of Sr; a yellowish-green flame-coloration shows that Ba is present. These flame-colorations should also be examined by the spectroscope (113).

The following is a less rapid method of detecting Ba and Sr. Dry the insoluble residue; fuse it on platinum foil with three or four times as much fusion-mixture, allow it to cool, then boil the mass with water until it is disintegrated; filter, wash the residue well upon the filter, and examine it by Table IV. (1031) for Ba and Sr only.

1018. Mn and Zn may accompany the precipitate of $Fe_2(HO)_6$, $Al_2(HO)_6$, $Cr_2(HO)_6$ in Group III.A. The precipitation of Mn is partly prevented by keeping the liquid from exposure to the air after AmHO has been added in excess. But the further precaution should be taken of dissolving the precipitate, which has been produced by AmHO, in HCl, and reprecipitating it by AmHO: the liquid is then filtered, and the filtrate is added to the filtrate which was originally separated from the Group III.A. precipitate. This treatment of the precipitate should be repeated several times, if small quantities of Zn or Mn have to be looked for in the presence of Al or Fe.

It is better still to precipitate Groups III A. and III.B. together (1058), and to examine the precipitate by Tables III.C., III.D. (1063, 1064) See rules given in paragraph 1062.

1019. The filtrate or solution, after the addition of AmCl and AmHO, may be coloured *blue* by Ni, *brown* by Co, or *reddish-violet* by $Cr_2H_6O_6$, which have been dissolved in the excess of AmHO.

The $Cr_2H_6O_6$ must be removed before proceeding to examine for the remaining Groups. It is readily separated, as a pale green flocculent precipitate, by boiling the liquid for a short time in a porcelain dish, a little more AmHO being added if the liquid does not smell of NH_3 after it has been boiled for a short time. This precipitate is filtered off, and the filtrate is further examined for Groups III.B., IV., and V. by the General Table.

The coloration of the ammoniacal liquid, which is caused by Ni or Co, is not removed by boiling the liquid.

1020. A *brown* or *dark colour* in the filtrate from the Group III.B. precipitate shows that Ni is present,* the colour being due to the solution of some of the NiS in the excess of yellow Am_2S. If the filtrate is brown or black, it should be poured into a porcelain dish and boiled until black NiS settles down and leaves a colourless liquid, when the lamp is removed for a few minutes it may be necessary to add a little water, in order to prevent evaporation to dryness. The liquid is then passed through a small filter, and the black precipitate is tested for Ni by fusing the portion of the filter-paper, which is stained by the NiS, into a colourless borax-bead. If the bead is *violet* or *brown* while hot and *yellow* when cold after it has been heated in the outer flame, and *becomes grey or opaque* after it has been heated in the inner flame, Ni is certainly present, and need not be further tested for in Table III B. (1028).

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* If Groups III.A. and III.B. have been precipitated together by AmCl, AmHO and Am_2S added in succession (1058), the coloration

The colourless filtrate from which the NiS has been precipitated, is examined by the General Table for Groups IV. and V.

1021. Part of the precipitate, which has been produced by Am_2CO_3, often adheres firmly to the inside of the vessel in which it has been produced. If this is the case, the tube should be rinsed out several times with distilled water, and the precipitate dissolved by pouring in some hot $H\bar{A}$ and causing it to run over the inside of the tube. This solution is added to the $H\bar{A}$-solution of the Am_2CO_3-precipitate, which is subsequently prepared for Table IV. (1031).

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of the filtrate may also be due to $Cr_2H_6O_6$. When the filtrate which is coloured by $Cr_2H_6O_6$ is boiled in a porcelain dish for several minutes, *pale green* $Cr_2H_6O_6$ is precipitated, which cannot be mistaken for *black* NiS.

1022. TABLE I.—SILVER GROUP.

The precipitate produced by HCl may consist of $PbCl_2$, AgCl, and Hg'_2Cl_2: the precipitate on the filter is washed five or six times with boiling water, the first portions of the washings being kept apart :—

The first portions of the washings may contain $PbCl_2$ in solution if this is present in any quantity, it will be deposited in brilliant crystals as the liquid cools. Test for, or confirm the presence of Pb, by adding to the washings K_2CrO_4: a yellow precipitate, readily soluble in KHO, shows :— *Presence of Pb.*	*Residue:* the residue is rinsed into a test-tube (100 *a*, *f*) and boiled with AmHO, and the liquid is then filtered :—	
	Filtrate add HNO_3 until the liquid is acid · a white precipitate, which unites into curdy particles when the liquid is shaken or boiled, shows :— *Presence of Ag.*	*Residue* on the filter is black: dry at a gentle heat, scrape the precipitate off the filter, mix with some dry Na_2CO_3, and heat in a small ignition tube (282). Globules of Hg are seen on the sides of the tube, but often only when the inside of the tube is rubbed with a rod or examined with a lens:— *Presence of Hg'* (Mercurosum).

1023. TABLE G. — EXAMINATION OF THE PRECIPITATE FORMED BY BOILING THE HCl SOLUTION WITH H_2SO_3. (See par. 1012)

The precipitate produced by H_2SO_3 may contain Au, $PbSO_4$, $BaSO_4$, and $SrSO_4$; the last three substances are perfectly white, and Au, if present, is therefore usually seen by colouring the precipitate brown or black; the Au is also usually visible during precipitation by H_2SO_3, or after the white sulphates have subsided, since it colours the liquid blue by transmitted and reddish by reflected light.

Ba and Sr can often be at once detected, if present, by taking a little of the moist precipitate on a loop of platinum wire, heating it in the inner blowpipe-flame for some time, moistening with HCl, and examining by the flame-coloration test. A more trustworthy method of proceeding is the following.

Rinse the precipitate into a small porcelain dish, using as little water as possible; dissolve in the liquid a small crystal of $H_2\bar{T}$ or add a few drops of strong $H\bar{A}$; then add $AmHO$ until it is just in excess, and boil for a short time; let the liquid stand for a short time, and decant through a filter:—

<table>
<tr>
<td rowspan="2"><i>Solution</i> may contain $PbSO_4$, add $H\bar{A}$ in excess, then K_2CrO_4, a yellow precipitate soluble in KHO shows —
<i>Presence of Pb.</i></td>
<td colspan="2"><i>Residue:</i> pour upon the residue in the dish a little HCl and a few drops of HNO_3, heat to boiling, let stand, and decant into a porcelain dish :—</td>
</tr>
<tr>
<td><i>Solution</i> boil until the smell of Cl is removed, add several drops of fresh $FeSO_4$ solution, a blue coloration and reddish precipitate show.—
<i>Presence of Au.</i></td>
<td><i>Residue:</i> dry by gently heating the dish, fuse with fusion mixture on platinum foil, and examine the mass for Ba and Sr as directed in the last part of 1017.</td>
</tr>
</table>

These lines run across both pages.

1024. TABLE II.—COPPER AND

The precipitate produced by H_2S in the HCl solution, after having with a small quantity of yellow Am_2S*, or with much KHO solu-

GROUP II A.—COPPER GROUP.

<table>
<tr><td colspan="5">1025 <i>The residue</i> may contain HgS, PbS, Bi_2S_3, CuS, CdS After having washed the precipitate and allowed it to drain, remove it from the filter into a porcelain dish(100<i>d</i>), pour upon it strong HNO_3 sufficient to cover it completely, and heat gently as long as any red fumes come off, adding more strong HNO_3 if necessary to prevent evaporation to dryness Boil off nearly all the acid, add a little dilute H_2SO_4, let stand for some time, stirring occasionally, and filter .—</td></tr>
<tr><td colspan="2" rowspan="2"><i>Residue</i> may contain HgS (<i>black</i>), $PbSO_4$ (<i>white</i>); but if <i>white</i>, the absence of Hg is not proved.
Remove the residue into a porcelain dish, using as little water as possible, add a little $H_2\bar{T}$, then AmHO in excess, boil and filter —</td><td colspan="3"><i>Filtrate</i> may contain Bi, Cu, Cd
Add AmHO in excess, boil and filter :—</td></tr>
<tr><td rowspan="2"><i>Precipitate</i> (not easily seen in dark blue solutions) wash with hot water, dissolve off the filter by pouring upon it <i>a few drops</i> of boiling dilute HCl, let this solution drop into a large quantity of cold distilled water, a milkiness appearing at once or after stirring and letting stand for some time, shows — <i>Presence of Bi</i>.
———
<i>Note</i> — A precipitate produced by AmHO must always be tested as above for Bi, since Pb and Hg might possibly appear here as white precipitates and be mistaken for Bi.</td><td colspan="2"><i>Filtrate, if blue, contains Cu</i>. (<i>Note</i> 2). add HCl until the liquid is acid, then pass H_2S to saturation, filter and wash quickly, keeping the filter covered with a glass plate, rinse the precipitate into a test-tube, pour upon it some dilute H_2SO_4, boil and filter through a covered filter (note 3).—</td></tr>
<tr><td><i>Residue</i> · dry on the filter at a gentle heat, best in the steam oven. Scrape the dried residue off the filter (<i>Note</i> 1), and heat it, mixed with dry Na_2CO_3, in a small ignition-tube ; globules of Hg form on the sides of the tube, which become visible when rubbed or when looked for by a lens :— <i>Presence of</i> Hg'' (Mercuricum).</td><td><i>Filtrate</i> · Add $H\bar{A}$ in excess, then K_2CrO_4, a yellow precipitate — <i>Presence of Pb</i>.</td><td><i>Residue</i> dissolve by pouring a little boiling HNO_3 upon the filter, add to the solution AmHO in excess, then $H\bar{A}$ in excess and a few drops of K_4FeCy_6 a chocolate - red precipitate shows — <i>Presence of Cu</i>.</td><td><i>Filtrate</i> add much water and pass H_2S for some time, or add much H_2S-water, a yellow precipitate shows — <i>Presence of Cd</i>.
<i>Note</i> — If this precipitate is dark - coloured, CuS is present (see <i>Note</i> 3): filter it off, boil it once more with H_2SO_4, filter and test the filtrate by H_2S as above, for Cd.</td></tr>
</table>

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* If traces of Cu have to be tested for, Na_2S should be substituted for Am_2S, since CuS is

Note 1.—If the residue is too small to be removed, the portion of the paper containing it may be cut up and mixed with Na_2CO_3. For the precautions requisite in performing this test refer to paragraph **282**.

Note 2.—Another method of detecting Cd *in the presence of Cu*, consists in adding KCy solution to the blue ammoniacal liquid, and passing H_2S ; Cd if present falls as yellow sulphide ; Cu may then be tested for in the filtrate, which has been boiled down considerably with excess of HNO_3, by adding AmHO in excess, then $H\bar{A}$ in excess, and K_4FeCy_6 (**298**).

Note 3.—Unless this precipitate is filtered without delay in a covered funnel, washed quickly, and examined at once, CuS is liable to be oxidised and to pass into the filtrate as $CuSO_4$; it then discolours the CdS precipitate, or may

ARSENIC GROUPS.

been washed with hot water, is removed from the filter and boiled tion, and filtered :—

GROUP II.B.—ARSENIC GROUP.

The filtrate may contain As_2S_3, Sb_2S_3, SnS_2, SnS, [Au_2S_3, PtS_2]. Add to it HCl until it is acid, and pass H_2S unless the liquid smells strongly; the above sulphides if present will be reprecipitated; a mere milkiness due to precipitation of S may be disregarded. (*Note* 4.)

The precipitate is filtered off and examined as directed below (**1026**). See also *Note* 5.

1026. *The precipitate* is allowed to drain for some time upon the filter in the funnel. It is best then to drain it still further by carefully taking the filter out of the funnel, opening it out, and spreading it upon a pile of three or four cut filter-papers. The precipitate is then removed to a small porcelain dish (**100**, *d*, *f*) and heated for some time nearly to boiling with strong fuming HCl. The liquid is then cooled and filtered :—

The residue will consist of As_2S_3, which is almost insoluble in strong HCl. Dry the washed residue on the filter at a gentle heat, then mix it with three or four times as much powdered KCy and freshly dried Na_2CO_3, and heat the mixture in a small bulb-tube, removing any drops of water inside the tube by a twisted piece of filter-paper, a black mirror (**320**).—*Presence of As.* (Refer to *Note* 7.) *Note.*—The As_2S_3 may also be dissolved by heating it with a little fuming HNO_3. The excess of HNO_3 is boiled off and As is detected as H_3AsO_4 by adding AmCl, excess of AmHO and $MgSO_4$ (**326**); or by addition of $AgNO_3$, and then cautiously neutralising with AmHO (**323**).	*The filtrate* may be examined for Sb and Sn by either I. or II. below :— I. Place a piece of platinum-foil in a porcelain dish and pour the acid filtrate upon it, then touch the foil with a piece of Zn; H will come off with effervescence, and if either at once or after a few minutes a black stain appears upon the platinum the *presence of Sb* is indicated. Confirm as directed in *Note* 6. The platinum-foil is removed and pieces of Zn are placed in the liquid as soon as the bubbles of H cease to be given off, Zn still remaining undissolved, remove the pieces of Zn, rubbing and rinsing any dark deposit back into the dish (**338**); let this deposit settle, decant the liquid and heat the solid deposit with strong HCl for several minutes in a test-tube, dilute with a little water, filter if necessary, and add a few drops of $HgCl_2$ solution: a white or grey precipitate (**341**) indicates :—*Presence of Sn* (*Note* 7). Refer to **1072**, **1073**, if Au and Pt have to be tested for. II. The acid filtrate is poured into a little hydrogen flask (fig. 71, par. **316**), in which H has been coming off briskly for about five minutes, being produced by the action of a little dilute HCl upon some pieces of Zn. The H is lighted at the jet, and the inside of a small porcelain dish or crucible lid is pressed down upon the flame: a black stain, which is not dissolved by solution of bleaching-powder, shows —*Presence of Sb.* The residue in the flask is tested for Sn, as directed in the latter part of I. (above).

omewhat soluble in Am_2S; but Sn″S and Au_2S_3 are much more readily soluble in Am_2S.

be entirely overlooked if present in small quantity. It is well to pour a few drops of H_2S water into the funnel with each fresh addition of washing water.

Note 4.—*White* S will always be precipitated here, if *yellow* ammonium sulphide has been used above. Traces of the sulphides are detected by shaking up a part of the white liquid with benzene. The coloured sulphide is seen at the surface of contact of the benzene with the acid liquid.

Note 5.—The detection of As, Sb, Sn by the method given in **1026** is simple and rapid. It is excelled in delicacy and trustworthiness by the methods given in paragraphs **346** and **347**; but these methods need not be resorted to unless Au or Pt, or traces of As, Sb, Sn have to be tested for.

Note 6.—The Sb deposit on the Pt may, after rinsing the foil, be dissolved by heating the Pt in a test-tube with a little very dilute HNO_3. On cooling this solution, diluting it and passing H_2S, an orange-red precipitate will form either at once, or after the liquid has stood for some hours, confirming the *Presence of Sb.*

Note 7.—The passage of H_2S into the HCl solution in the General Table (1007) may have already indicated by the colour of the sulphide precipitate, or by precipitation occurring only on application of heat, whether As and Sn were originally present in the *-ous* or *-ic* condition. If this is not certain, tests 323-326 and 340–342 must be tried with the original solution of the substance.

1027. TABLE III.A.—IRON GROUP.

The precipitate produced by boiling, after addition of AmCl and of AmHO in excess, may contain $Al_2(HO)_6$, $Cr_2(HO)_6$, $Fe_2(HO)_6$; the colour of the precipitate will usually show whether it contains any quantity of $Fe_2(HO)_6$ (reddish-brown), or $Cr_2(HO)_6$ (pale green), since $Al_2(HO)_6$ is colourless.

Dissolve the precipitate in a *little* boiling dilute HCl (**100, d, e, f**), add to the solution pure KHO or NaHO until the precipitate remains after the liquid has been stirred, then add KHO or NaHO in considerable excess, boil for some time and filter :—

<table>
<tr>
<td rowspan="2">1. <i>Filtrate</i> may contain $Al_2(HO)_6$ dissolved in excess of NaHO : if it is green, $Cr_2(HO)_6$ is also in solution and must be precipitated by further boiling in a porcelain dish, and then be removed by filtration.
Add HCl in slight excess, then AmHO in slight excess (103) : a colourless gelatinous precipitate shows .—
<i>Presence of Al.</i>
<i>Note.</i> — Sodium and potassium hydrates are very liable to contain $Al_2(HO)_6$; the analyst must ascertain that the Al does not come from this source.</td>
<td colspan="2">2 <i>Precipitate</i> may contain $Cr_2(HO)_6$ and $Fe_2(HO)_6$.
Dry upon the filter, carefully avoiding charring the paper, when sufficiently dried the substance will have shrunk considerably into small, hard, dark-coloured pieces; detach these from the filter and fuse them with a little solid Na_2CO_3 and KNO_3 upon a piece of platinum foil, either supported upon a pipeclay triangle or held in the Bunsen flame by crucible tongs Continue the fusion by heating the under surface of the foil in the blowpipe flame as long as any frothing occurs, then allow to cool, a yellow mass indicates the presence of Cr (note 1) Boil the platinum foil in a small porcelain dish with water until the substance is entirely dissolved or only a dark brown powder (Fe_2O_3) remains undissolved, take out the foil, allow the powder to settle, and decant the liquid through a filter :—</td>
</tr>
<tr>
<td>3. <i>Filtrate</i> will be yellow if Cr is present (note 2).
Add $H\bar{A}$ in excess, shown by the colour becoming reddish, boil for several minutes, then add $Pb\bar{A}_2$ solution; a yellow precipitate shows :—
<i>Presence of Cr</i> (note 3).</td>
<td>4. <i>Residue</i> in porcelain dish; dissolve by pouring in a little HCl and boiling, add some water, then a few drops of KCyS solution; a blood-red coloration shows :—
<i>Presence of Fe</i> (note 4).</td>
</tr>
</table>

Note 1.—If Mn was present in the original substance, it is often precipitated more or less completely with the Fe, and will be detected by imparting to the fused mass a bluish-green colour. This should be noted, as the Mn may possibly be entirely precipitated in this Group, and will not then be detected in the next Group.

Note 2.—The green colour, due to the presence of Mn, conceals the pale yellow colour due to Cr. But when the cool mass is dissolved in water and the solution is boiled with $H\bar{A}$, the manganese coloration is destroyed. The yellow colour due to Cr then becomes evident, after any dark-coloured precipitate has been removed by filtration.

Note 3.—Cr may have been present as a chromic salt, in which Cr replaces the hydrogen of an acid; or as a chromate, in which Cr is united with oxygen to form an acid-radicle. Ascertain in which state Cr was present, by boiling some of the original substance with Na_2CO_3-solution and filtering. A yellow filtrate, which after it has been acidified with $H\bar{A}$ gives a yellow precipitate with $Pb\bar{A}_2$, shows that Cr was *present as a chromate.* If the Cr was previously combined with an acid-radicle as a chromic salt, it will remain undissolved as green $Cr_2(HO)_6$, and will not colour the filtrate · it is found by 2 in the preceding Table.

Note 4.—Fe is always detected here as a ferric salt · since, even if it was originally present as a ferrous compound, this will have been converted into a ferric salt, when the solution is boiled with HNO_3, before Group III.A. is precipitated in the General Table. In order to ascertain in which condition Fe was present, some of the original substance is boiled with HCl in a special apparatus (figs 40, 41, par 86), the solution is filtered, if necessary, and then divided into two parts, To one portion freshly made solution of K_3FeCy_6 is added (*Note,* 201), a dark blue precipitate shows the *Presence of Fe''* (*Ferrosum*), to another portion KCyS is added, a blood-red colour shows *Presence of Fe'''* (*Ferricum*)

1028. TABLE III.B.—ZINC GROUP.

The precipitate produced by Am_2S in the solution, or in the filtrate from Group III.A., may contain ZnS, MnS, NiS, CoS. The colour of the precipitate will usually show whether it contains any quantity of NiS or CoS which are black, or consists only of MnS (pink) or ZnS (white).

The presence of Ni will have been indicated by the blue colour of the filtrate, which was obtained after boiling with AmHO in the General Table, and by the dark brown colour of the filtrate which was obtained after boiling with yellow Am_2S. If the black precipitate, which was obtained by boiling the dark brown filtrate from Am_2S (1020), has been proved by the borax-bead to be NiS, the further tests for Ni in the following Table may be omitted.

Remove the precipitate from the filter with cold dilute HCl in the way described in par. **100 c**; take out the paper and allow the liquid to stand, occasionally stirring it well. The precipitate will either dissolve, leaving only white sulphur, in which case the milky liquid may be at once examined by **1030**; or a black residue will be left, which must be filtered off and examined by **1029**, the filtrate being examined by **1030**.

1029. *The residue* is black, and may contain NiS, CoS.

Examination by the borax bead

Fuse a small quantity in a clear borax bead in the extreme tip of the outer blowpipe flame, note its colour; then fuse the bead for some time in the inner blowpipe flame, and again observe its colour. Any one of the following observations may be made.—

Blue bead in both flames shows.— *Presence of Co.* Co need not be further tested for, but if any doubt exists as to the presence of Ni the rest of the residue must be examined as below.	*Brown or yellow bead,* when cold after fusing it in the outer flame, which becomes *grey or opaque* in the inner flame, shows *Presence of Ni, Absence of Co* Unless mere traces of Co have to be tested for, no further examination need be made	*A bead which is neither brown nor blue,* but of some intermediate hue on cooling from the outer flame, indicates the probable presence of both Co and Ni Examine the remainder of the black residue as directed below for Ni and Co, or for Co alone if Ni has been already detected by (1020).

Further examination of the residue.—Rinse the residue from the filter into a porcelain dish, using as little water as possible: pour in a little strong HCl and boil for some time, adding occasionally a small crystal of $KClO_3$, until the black residue is entirely dissolved or only a small quantity of dark sulphur remains, then boil down nearly to dryness, a blue liquid shows *presence of Co*; dilute with a little water, filter if necessary into a boiling-tube and pour in KCy solution slowly until the precipitate formed at first is just redissolved, boil briskly for several minutes, and add much strong NaOCl, or make decidedly alkaline with NaHO and add much Br-water; heat nearly to boiling, and allow the liquid to stand at least ten minutes, filter*:—

Precipitate (*black*): wash, and confirm the presence of Ni by fusing some of the precipitate, or the paper stained with the precipitate, in a borax bead in the outer and inner blowpipe flames a bead *yellow* when cold, becoming *black* in the inner flame shows.— *Presence of Ni.*	*Filtrate* must be warmed with more NaOCl, or Br-water, and filtered from any additional precipitate which may form, then evaporated to dryness and strong HNO_3 poured upon the residue as long as any frothing is caused, this is then evaporated just to dryness, the residue dissolved in water, excess of KHO added to it, and any precipitate filtered off and fused into a borax bead, taking the paper stained by the precipitate if the precipitate is not easily removed. a blue bead shows.— *Presence of Co.*

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* If this precipitate adheres to the sides of the boiling tube, it is dissolved, after the liquid has been emptied out, by pouring in a little boiling HCl; from this solution the Ni is precipitated by addition of KHO, and the precipitate is filtered off and tested by the borax bead for Ni.

1030. *The Solution or filtrate* may contain Zn, Mn.
Boil in a porcelain dish until it ceases to smell of H_2S, then drop in a small crystal of $KClO_3$, and boil for several minutes. After cooling the liquid add pure NaHO until after stirring the liquid it turns red litmus-paper blue, then add more NaHO, stir well and filter:

Precipitate is white at first, but rapidly darkens in the air. Fuse a portion of it with solid Na_2CO_3 and KNO_3 on platinum foil, a bluish green mass is obtained on cooling, showing *Presence of Mn.* *Note.*—Mn may have been originally present either replacing H in an acid, or combined with O as an acid-radicle. Its presence in an acid-radicle is indicated by the colour of the original solution, since in the form of manganate it is green, and as permanganate it is purple; these colours disappear on boiling the HCl solution for some time, or on passing H_2S.	*Filtrate;* pass H_2S, a white precipitate forms, often somewhat discoloured, showing: *Presence of Zn.* *Note.*—From a dilute solution of $Zn(HO)_2$ in NaHO the $Zn(HO)_2$ is precipitated on boiling, hence after adding NaHO, as directed above, the liquid must not be boiled, else Zn might be precipitated with the $Mn(HO)_2$ and escape detection. But by diluting and boiling the above filtrate, it is usually possible to cause any $Zn(HO)_2$ which it holds in solution to precipitate, especially if the alkalinity of the liquid is somewhat reduced by cautious addition of HCl in quantity insufficient to make the liquid acid. If this precipitate is separated by pouring the liquid through a double filter and is then dissolved off the filter and the sides of the boiling tube by a little boiling $H\bar{A}$, ZnS may be precipitated *perfectly white* from this solution by H_2S.

1031. TABLE IV.—BARIUM GROUP.

The precipitate, which has been produced by Am_2CO_3, may contain $BaCO_3$, $SrCO_3$, $CaCO_3$. It is to be well washed with boiling water. Before the whole of the precipitate is dissolved off the filter, much may usually be learnt by making an examination of the flame-coloration, which is yielded by the solution of a part of the precipitate in HCl (**1032**). The results thus obtained are afterwards confirmed in the wet way by par. **1033**, or by the quicker method in par. **1034**.

1032. *Examination by Flame-Coloration.*—Take a small quantity of the precipitate off the filter upon the end of a glass rod, and dissolve it by moving the end of the rod about in several drops of HCl on a watch-glass; dip into this solution a loop of platinum wire, which imparts no colour to the flame (**112**, Exp. 91), and hold the loop for some time in the Bunsen-flame: repeat these processes if the coloration has not been satisfactorily observed. Ca will impart to the flame a *yellowish-red* colour, Sr a *crimson-red*, Ba a *yellowish-green*. The red colorations yielded by Ca and Sr are distinguished by viewing the flame through the indigo-prism; the Ca

These lines run across both pages.

coloration then appears *dingy green*, while the Sr-coloration
remaining after the other colorations have disappeared.
are apt to interfere with one another; but if at any time a
present; if a green coloration is visible without using the
may also be examined by the spectroscope (113).

1033. *Examination in the Wet Way.*—Dissolve the rest
$H\bar{A}$; boil the liquid and pour it again through the filter, if
not acid in reaction, mix more $H\bar{A}$ with it, then pour off a
the larger portion (*B*). Cool portion (*A*) and add to it several
refer to column I. below, or to IV. (1034) if no immediate
cipitate then forms refer to II. or to V. (1034); if no
by III. or by VI. (1034).

<table>
<tr><td colspan="3">I. <i>An immediate precipitate is produced by $CaSO_4$ in the cold, showing Presence of Ba.</i>
Portion (<i>B</i>) must then be tested for Sr and Ca which may also be present: examine it as directed below :—
Add K_2CrO_4 solution until the colour of the liquid is reddish yellow; heat, and filter through a double filter-paper, pouring the filtrate through the same filter repeatedly if necessary until the liquid is quite clear; then add to the liquid, which must be orange red in colour, AmHO until the colour changes to pale yellow, then add Am_2CO_3 solution :—</td></tr>
<tr><td rowspan="2"><i>No precipitate forms,</i> showing :—
<i>Absence of Sr and Ca.</i></td><td colspan="2"><i>A precipitate forms,</i> showing Sr, Ca, or both of them, to be present. Add to the liquid Am_2CO_3 in excess, filter, reject the filtrate and dissolve the precipitate off the filter in as little boiling $H\bar{A}$ as possible; pour off a small part (<i>C</i>) of this solution, reserving the larger portion (<i>D</i>). To (<i>C</i>) add $CaSO_4$ solution and boil :—</td></tr>
<tr><td><i>No precipitate forms:—</i>
<i>Absence of Sr.</i>
Examine portion (<i>D</i>) for Ca as directed at (<i>D</i>) in Column III.</td><td><i>A precipitate forms:—</i>
<i>Presence of Sr.</i>
Examine portion (<i>D</i>) for Ca as directed at (<i>D</i>), Column II.</td></tr>
</table>

1034. BARIUM GROUP.—

IV. Add K_2CrO_4 solution to portion (*B*) until the liquid is coloured, heat, and filter through a double filter-paper.
The yellow precipitate confirms the presence of Ba.

For the examination of the Filtrate refer to Column V.

still appears *crimson;* Ba is usually found by its coloration
When all three metals are present together, their colorations
red colour is visible through the indigo-prism, Sr is certainly
prism, the presence of Ba is proved. The flame-coloration

of the precipitate off the filter by pouring in a little boiling
the precipitate is not entirely dissolved. If the solution is
small portion (*A*) of the solution into a test-tube, and put by
drops of $CaSO_4$-solution; if an immediate precipitate forms,
precipitate forms heat the liquid to boiling, and if a pre
precipitate forms even after several minutes, test portion (*B*)

II. *A precipitate is not formed at once on the addition of $CaSO_4$, but appears on boiling the liquid, showing:—*	III. *No precipitate is produced by $CaSO_4$, even on boiling, showing:—*
Absence of Ba and Presence of Sr. *D.* Portion (*B*) is then examined for Ca as directed below:— Add dilute H_2SO_4 in excess, boil and filter, reject the precipitate; add to the filtrate several drops of H_2SO_4 and boil; if this causes any precipitate boil and filter, and again test the filtrate by addition of H_2SO_4 and boiling: repeat this process if necessary. To the clear filtrate, which gives no further precipitate on addition of H_2SO_4 and boiling, add gradually AmHO until after mixing the liquid by thorough stirring or shaking, it turns red litmus-paper blue, then add $Am_2C_2O_4$ solution and warm gently; a white precipitate, often appearing only after a time, shows:— *Presence of Ca.* (See Note, Column III.)	*Absence of Ba and Sr, and Presence of Ca.* *D.* Confirm the presence of Ca by making portion (*B*) alkaline with AmHO (103), then add $Am_2C_2O_4$ solution; a white precipitate forms, showing:— *Presence of Ca.* *Note.*—If mere traces of Ca have to be tested for, the filter-paper used in the analysis must first be freed from any traces of Ca it may contain by wetting it with dilute HCl, and then thoroughly washing it with distilled water, as described in the last par. of 92.

TABLE IV.A.

V. *The Solution or Filtrate* may contain Sr and Ca.	VI. *The Solution or Filtrate* may contain Ca.
Dilute with twice its volume of water, add dilute H_2SO_4 in excess (978), boil and filter. ***The Precipitate*** is tested for Sr by moistening it with strong HCl and examining the flame-coloration (180) through the indigo-prism, or the spectroscope. ***The Filtrate*** is examined by Column VI.	Add AmOH in excess, then add $Am_2C_2O_4$, and allow to stand: a white precipitate:— ***Presence of Ca.***

1035. TABLE V.—POTASSIUM GROUP.

The filtrate, which remains after all the Group-reagents have been added in succession to the original solution, may still contain Mg, K, Na, and NH_4. Since, however, NH_4-salts have been added as Group-reagents, NH_4 must be tested for in the original substance by KHO solution (989, I, 3). Proceed to examine for other members of Group V. as is directed below.

Evaporate the filtrate from Group IV. to dryness in a porcelain dish, scrape out the solid residue, and heat it to redness upon a piece of platinum foil in the Bunsen-flame, as long as any white fumes are seen to be given off when the foil is removed for an instant from the flame (*Note* 1). All NH_4-compounds are thus entirely removed. If any residue remains (*Note* 2), it is dissolved by boiling the foil in a test-tube with a small quantity of water, to which several drops of dilute HCl have been added. Divide this solution into two parts:—

<table>
<tr><th>EXAMINATION FOR Mg.</th><th colspan="2">EXAMINATION FOR K AND Na.</th></tr>
<tr><td rowspan="3">Before testing for Mg in one portion of this solution, it is necessary to remove from it any traces of Ba, Sr, or Ca which it may possibly contain,* and which might else be mistaken for Mg.
Add, therefore, several drops of H_2SO_4 to the liquid, boil for a short time and let stand; then, whether a precipitate has formed or not, add a little AmCl, then AmHO in excess, then several drops of $Am_2C_2O_4$, and warm gently; if any precipitate has formed proceed to filter at once as quickly as possible. To the liquid, in which the addition of H_2SO_4, AmCl, AmHO in excess and $Am_2C_2O_4$ causes no further precipitate, add Na_2HPO_4, and if no precipitate forms at once warm gently, shake or stir the liquid violently and let it stand for some time: a white crystalline precipitate shows:—
Presence of Mg.</td><td colspan="2">In the other portion proceed to test for K and Na as directed below The presence of Mg in no way interferes with the detection of K and Na and hence if Mg has been detected its removal is unnecessary
Flame coloration—Dip into the solution a loop of platinum wire which has been proved not to impart any colour to the flame Hold the loop in the Bunsen flame, one of the following results will be observed.—</td></tr>
<tr><td>*A bright yellow flame coloration,* indicating the *presence of Na* (Note 3)
Examine this coloration through the indigo-prism; if it appears red, *presence of K* is proved if no red colour is visible K is probably absent, or is present only in very minute quantity.</td><td>*A pale lilac coloration,* appearing crimson red through the indigo-prism, shows —
Presence of K, and absence of Na.</td></tr>
<tr><td colspan="2">It is usual to confirm the results of the examination of the flame coloration by pouring the remainder of the solution upon a watch glass, adding to it several drops of $PtCl_4$ (Note 4) and stirring well for some time; the formation of a yellow precipitate shows:—
Presence of K.
See (141), and Note 5, below.</td></tr>
</table>

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* Traces of Ba, Sr, Ca may remain unprecipitated by Am_2CO_3.

Note 1.—If the residue is small in amount, it may be ignited in the dish; but this is not to be generally recommended, since the porcelain dish is liable to be cracked by the heat, and it is also difficult to get entirely rid of the NH_4-salts by heating the residue in porcelain.

Note 2.—It is not safe to place much reliance upon an examination of the foil for the detection of a small quantity of residue, but it may usually be detected by producing a crackling noise while the foil is cooling immediately after its removal from the flame. Should there be any doubt, the foil must be boiled with water and a drop of HCl, and the solution examined for Mg, K, and Na, as is directed above.

Note 3.—The examination of this flame-coloration by means of the spectroscope (113) will naturally suggest itself

A yellow coloration, more or less intense, will almost always be obtained here, since most substances and reagents contain small quantities of Na. Hence the student must note the intensity of the coloration, and judge from it whether the quantity of Na is small or large. The test for a trace of Na is usually made upon the original substance

Note 4.—If iodine is present, $PtCl_4$ will produce an intense red coloration. Hence if iodine is suspected to be present, the above solution should be evaporated to dryness with a little strong HNO_3, and the residue dissolved in a few drops of dilute HCl before it is tested for K with $PtCl_4$. This is of course unnecessary, if the solution has already been evaporated to dryness with HNO_3 before precipitating Group III. in the General Table In case of uncertainty, add $PtCl_4$ to a drop only of the above solution, and if iodine is found to be present, proceed as is directed above.

Note 5.—Mere traces of K and Na may be detected by adding $PtCl_4$, and then evaporating the liquid to dryness in a porcelain dish upon a water-bath Absolute alcohol is then poured into the dish and stirred. Any yellow residue shows the *Presence of K*. The solution will give the pure Na-coloration, if Na is present. and if the yellow residue is filtered off and washed with absolute alcohol, it will yield the pure lilac flame-coloration of K.

EXAMINATION FOR THE RARER METALS, AND FOR ORGANIC SUBSTANCES.

1037. The Systematic Examination for the more commonly occurring metals is now complete. If the Rarer Metals have to be looked for, attention must be paid to the special Tables given in paragraphs **1090, 1091.**

The detection of Organic Substances is provided for in paragraphs **1092,** *et seq.*

1038. TABLE OF SOLUBILITY.

A blank signifies that the solubility is unknown or unimportant.

	K.	*Na*	*Am*	*Mg*	*Ba*	*Sr.*	*Ca.*	*Fe″.*	*Fe‴.*	*Al.*	*Cr.*	*Zn.*	*Mn*	*Ni.*	*Co.*
Oxide . .	w.	w.	w.	a	w	w.	w-a.	a.	a.	a i	a i.	a.	a.	a.	a.
Sulphide . .	w.	w.	w	w-a	w	w	w-a	a^{m}.	a.	—	—	a.	a.	a^{mn}	a^{mn}.
Chloride . .	w.	w.	w.	w.	w.	w	w	w	w.	w.	a i.	w.	w.	w.	w.
Iodide . . .	w.	w	w	w	w.	w	w.	w	w.	—	—	w.	w	—	—
Sulphate . .	w.	w.	w	w	i	i	w-a^{m} -i	w	w.	w	w a	w.	w.	w.	w.
Nitrate . .	w.	w.	w.	w	w	w	w	w.	w.	w	w.	w.	w.	w.	w.
Phosphate .	w.	w	w	a	a	a	a	a	a.	a	a	a.	a	a.	a.
Carbonate .	w.	w.	w.	a.	a.	a.	a	a.	—	—	—	a.	a.	a.	a.
Borate . . .	w	w	w	a.	a.	a	a.	a.	a.	a	a	a.	a.	a.	a.
Arsenite . .	w.	w.	w.	a.	a	a	a.	a	a.	a	a.	a.	a.	a.	a.
Arsenate . .	w.	w	w	a.	a.	a	a.	a	a.	—	—	—	—	a.	a.
Chromate . .	w.	w	w	w.	a.	a.	a	—	w.	a.	a	w	i.	a.	—
Fluoride . .	w	w.	w.	a	a i	i.	a-i.	—	—	—	—	—	—	—	—
Oxalate . .	w.	w	w.	a	a.	a.	a.	a.	a.	a	w-a	a.	w-a	a.	a.

	Hg″	*Pb*	*Bi.*	*Cu.*	*Cd.*	*Sb.*	*Sn″.*	*Sn⁗.*	*As″.*	*Ag.*	*Hg′.*
Oxide . . .	a	a^{n}	a.	a.	a.	a^{m}.	a	a i.	w a^{m}	a^{n}.	a^{n}.
Sulphide . .	a^{mn}	a^{n}	a^{n}	a^{n}	a.	a^{m}	a^{m}	a^{m}	a^{n}	a^{n}.	a^{n}.
Chloride . .	w.	w i	w b.	w.	w	w b	w b.	w.	w.	i.	a i.
Iodide . . .	a	w a^{m}.	—	—	w.	—	w	w	—	i	a^{n}.
Sulphate . .	w b	a i.	w b	w.	w.	a.	w.	w b.	—	w.a^{n}	w.b.a^{n}
Nitrate . .	w b	w	w b	w.	w	—	—	w	—	w.	w.b.
Phosphate .	a.	a^{n}	—	a	a	—	a	—	—	a^{n}	a^{n}
Carbonate .	a.	a^{n}.	a.	a.	a	—	—	—	—	a^{n}.	a^{n}.
Borate . . .	—	a^{n}	a.	a	w a.	—	a.	—	—	—	w.
Arsenite . .	a.	a^{n}	a	a.	—	a.	—	—	—	a^{n}	a^{n}.
Arsenate . .	a.	a^{n}	—	a	—	a.	—	—	—	a^{n}.	a^{n}.
Chromate . .	w-a	a^{n} i	a.	w.	—	a.	—	—	—	a^{n}.	a^{n}.
Fluoride . .	w	a	—	—	—	—	—	—	—	w.	—
Oxalate . .	a	a	a.	a.	a.	a.	a.	w.	—	a.	a.

Refer to par. 1040. The solubility of a compound is denoted by letters ;—

w. Signifies soluble in water.

a. Soluble in the acids HCl, HNO_3, and aqua regia.

a^{m}. Soluble in muriatic, or hydrochloric acid.

a^{n}. Soluble in nitric acid.

a^{mn}. Soluble in a mixture of muriatic and nitric acids, or aqua regia, but not in either acid separately.

i. Insoluble in water and in acids.

w.a. Letters thus placed together, with a stop between them, signify that in different states the substance shows the different solubilities denoted by the letters.

w-a. Letters connected by a hyphen indicate that the substance is only slightly soluble in the first solvent, and may therefore partially fall under the class denoted by the second letter.

w.b. Decomposed more or less by much water with formation of a basic salt, which is insoluble in water but soluble in acid.

EXAMINATION FOR ACID-RADICLES.

Introductory Remarks.

1039. In conducting the Examination for Acid-radicles, much time and trouble may usually be saved by considering which of these radicles can possibly be present. The preceding examination will usually have limited the number considerably (**1041**); but it may be further reduced by a consideration of the metals present and of the solubility of the substance under analysis, aided by a reference to the Table of Solubility (**1038**).

1040. Table of Solubility.—The Table on the preceding page has been drawn out to assist the analyst in the above consideration.

Only commonly occurring compounds are contained in this table; the solubility of other substances may be obtained by reference to a *Dictionary of Solubilities.*

The Table is thus arranged. In a horizontal line at the head are placed the symbols of the more commonly occurring metals, which yield salts or basic oxides. In the vertical column on the left is a list of that portion of the names of the compounds of these metals, which corresponds to the acid-radicle.

To find the Solubility of a Compound of any one of the metals which are placed at the top of the Table, glance down the vertical column which is headed by the metal. The letter which indicates the solubility of the compound will be found in a horizontal line with the acid-radicle portion of the name of the compound. Thus, in order to find the solubility of zinc sulphate, it is only necessary to glance down the vertical column with *Zn* at its head: on a horizontal line with *sulphate* stands the letter *w*; this indicates that zinc sulphate is soluble in water.

The Way in which this Table is used, after the solubility of the substance under analysis has been ascertained, and after the metals which it contains have been detected, may be explained by an example.

In a substance, *which was entirely soluble in water*, the metals K, Ba, Ag were found. A glance down the columns, headed by these three metals, shows at once which acid-radicles may be present.

All acid-radicles might be present combined with K, since all its salts are soluble in water. But the presence of Ba in a substance which is soluble in water, shows that SO_4, PO_4, CO_3, BO_3, AsO_4, CrO_4, and F cannot be present; since these acid-radicles form compounds with Ba which are insoluble in water. Ag excludes in addition S, Cl, Br and I. Hence, of the more commonly occurring acid-radicles, NO_3 only need be tested for.

This example shows how much the examination for acid-radicles may often be simplified, when the metals which are present in a substance and the solubility of the substance are known.

1041. Several Acid-radicles will probably have been satisfactorily tested for in the Preliminary Examinations. In case their reactions have been interfered with by the presence of other substances, the analyst should refer to the reactions for acid-radicles in the Fourth Section (Part III.), and select one which will be decisive.

The Examination for Metals may also have yielded proof of the presence of certain acid-radicles. Thus:

A green alkaline solution, becoming purple when it is diluted or acidified, shows . . . . *Presence of* $(MnO_4)''$

A reddish-yellow solution, becoming green and depositing sulphur when H_2S is passed, shows . .	*Presence of* $(CrO_4)''$
A purple solution, becoming colourless with H_2S, shows . . .	*Presence of* $(MnO_4)'$
A yellow precipitate of As_2S_3 with H_2S, which appears only when the liquid is boiled, shows . .	*Presence of* $(AsO_4)'''$
An insoluble residue of SiO_2, after the filtrate from the H_2S-group has been evaporated to dryness, shows	*Presence of* $(SiO_3)''$
And a yellow precipitate obtained with $AmHMoO_4$, before precipitating Group III., shows . .	*Presence of* $(PO_4)'''$

If the presence of any of these acid-radicles has been detected in the above manner, it will require no further confirmation.

Of the Acid-radicles which remain to be tested for, some can be detected by the plan drawn out in paragraphs **1042–1047**, others are most easily found by applying special tests to the original substance (**1048–1056**).

GENERAL EXAMINATION FOR ACID-RADICLES.

1042. Removal of Metals.—Before the following tests are applied, it is advisable to remove from the substance any metals, other than K, Na and NH_4, which it may contain, since some of these might be precipitated by the reagents which are added for the detection of the acid-radicles. If alkali-metals alone are present, this separation is unnecessary, since they are not precipitated by any of the reagents.

This removal of the metals may usually be effected by boiling a portion of the finely-powdered substance with pure

Na_2CO_3-solution, which must be added to the clear solution as long as it causes any precipitate. The precipitated carbonates are then removed by filtration, and the clear filtrate is divided into five equal portions. One of these portions is reserved in case of accident, and a second is kept for the tests for organic acid-radicles. The other three portions are acidified, while they are hot, by the addition of HCl, HNO_3 and $H\bar{A}$ respectively; they are then examined as is directed below, a separate part being used for each test.

1043. **Some Metals cannot be Completely Precipitated as Carbonates** by boiling the substance with Na_2CO_3-solution . the presence of these metals in the solution is, however, frequently of no consequence. If they must be precipitated, the addition of Am_2S, or the passage of H_2S, will usually separate them as insoluble sulphides ; but the subsequent separation of the excess of Am_2S or H_2S, by gently warming the solution, is troublesome.

Since the analyst knows at this stage of the analysis what metals are present, it should not be difficult to separate them, or to allow for their presence while the tests for acid-radicles are being tried.

SPECIAL TESTS FOR ACID-RADICLES IN SOLUTION.

1044. **The Three clear Acidified Portions** of the Filtrate, obtained by boiling the substance with sodium carbonate-solution (1042), are tested by paragraphs 1045–1047. Further special tests (1048–1056) are then tried, if necessary.

Some general tests for organic acid-radicles will be found in paragraphs 1103–1106.

1045.	I. *Portion acidified with HCl.*	*Present.*
	On addition of $BaCl_2$ solution a white precipitate insoluble on boiling (*Note* 1)	$(SO_4)''$
	On addition of $BaCl_2$ solution, a semi-transparent precipitate insoluble on boiling (*Note* 2) . .	$(SiF_6)''$
	On addition of AmCl and Am_2CO_3, a semi-transparent precipitate	$(SiO_3)''$
	On addition of $FeSO_4$ solution, a dark blue precipitate	$(FeCy_6)'''$
	On addition of Fe_2Cl_6 solution, a dark blue precipitate: $FeSO_4$ yielding a light blue precipitate .	$(FeCy_6)^{iv}$
	On addition of Fe_2Cl_6 solution, a red coloration, destroyed by pouring into $HgCl_2$ solution . .	$(CyS)'$

1046.	II. *Portion acidified with* HNO_3. Refer to (*Note 3*).	*Present.*
	On addition of $AgNO_3$, a *pure white* precipitate, easily soluble in AmHO (*Note* 1)	Cl'
	On addition of $AgNO_3$, a *light yellow* precipitate, with difficulty soluble in AmHO	Br'
	On addition of $AgNO_3$, a *yellow* precipitate, almost insoluble in AmHO	I'
	Refer to (*Note* 4)	

1047.	III. *Portion acidified with* $H\bar{A}$	*Present.*
	On addition of $Pb\bar{A}_2$ solution a yellow precipitate (*Note* 5)	$(CrO_4)''$
	On addition of $CaCl_2$ solution a white gelatinous precipitate	F'[*], probably.
	On addition of $CaCl_2$ a white pulverulent precipitate	$(C_2O_4)''$, probably.
	On addition of Fe_2Cl_6 a yellowish white precipitate (1052, 1053)	$(PO_4)'''$, or $(AsO_4)'''$.

NOTES ON THE PRECEDING TABLE.

Note 1. Unless the Na_2CO_3-solution, which was used in preparing the solution for these tests, was pure, (SO_4) and (Cl), if detected, may have been present only as impurities in the Na_2CO_3 and not in the original substance; portions of the original substance should then be tested by paragraphs 1048 and 1049.

Note 2 The presence of (SiF_6) should be confirmed by adding KCl to another part of the solution (611), or by heating the $BaSiF_6$ precipitate or the original substance with strong H_2SO_4 (612).

Note 3. If $AgNO_3$ yields a black precipitate, this proves the presence of a sulphide, or possibly of a thiosulphate. Add HNO_3 and boil; the black Ag_2S will thus be decomposed, leaving a milky liquid, in which any other precipitate is readily seen after it has been coagulated by heating or shaking the liquid.

Note 4. It must be remembered that (Cy), $(FeCy_6)_{iv}$, $(FeCy_6)'''$ and (CyS) are also precipitated by $AgNO_3$. Accordingly, if these acid-radicles have been already found, a precipitate produced by $AgNO_3$ does not prove the presence of (Cl), (Br), or (I). These acid-radicles must therefore be specially examined for, as is directed below.

If chloride, bromide, and iodide have all to be tested for, a portion of the Na_2CO_3-solution must be examined by paragraph 572 or 573; or the precipitate obtained by $AgNO_3$ (1046) may be tested by paragraph 571 for Cl, Br, I. If only bromide and iodide are to be tested for, use paragraph 574.

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[*] F will be readily detected in this precipitate, or better in the original substance, by paragraph 1055.

Note 5. If a white precipitate of $PbSO_4$ is produced here, it may be dissolved by adding AmHO in excess and warming the liquid (286); red basic lead chromate will remain, if a chromate was present.

FURTHER SPECIAL TESTS FOR ACID-RADICLES.

1048. **Sulphate.**—A portion of the original substance is boiled with HCl, and the liquid is decanted or filtered, if necessary, and is tested with $BaCl_2$; a white precipitate shows the *Presence of* SO_4.

1049. **Chloride.**—A portion of the original substance is warmed with HNO_3, and the solution is decanted or filtered, if necessary, and is tested by $AgNO_3$; a *perfectly white* precipitate, which is easily soluble in warm AmHO, shows the *Presence of Chloride.*

1050. **Nitrate.**—The clear aqueous solution or extract of the substance is mixed with its own volume of strong H_2SO_4, and the liquid is cooled. Freshly-made cold solution of $FeSO_4$ is poured upon the surface of the acid liquid without mixing; a brown layer or ring at the surface of contact of the liquids (537) shows the *Presence of* NO_3.

Note.—If nitrite is present, it must be removed by boiling the substance with excess of $H\bar{A}$ before the above test is applied.

1051. **Cyanide,** if present, will have been detected by the special test (983), and by the smell of bitter almonds, given off by the substance after H_2SO_4 has been added (997, Exp. 1). Ascertain in what form cyanogen is present by paragraph 1084.

1052. **Arsenate.**—This acid-radicle cannot be present unless As has been detected during the examination for metals. The presence of (AsO_4) is rendered probable, by the precipitation of yellow As_2S_3 occurring only when the acid liquid is boiled after it has been saturated with H_2S (1016).

If As has been found amongst the metals, proceed to test for (AsO_4) by adding HCl to a portion of the Na_2CO_3-solution (1042) until it is acid, then heating to expel CO_2, and adding AmCl, AmHO in excess, and $MgSO_4$. Filter off any precipitate which forms on warming and shaking the liquid, and pour a few drops of $AgNO_3$-solution upon the white precipitate on the filter; a change of colour to brown shows the *Presence of* AsO_4.

1053. **Phosphate.**—Boil some of the original substance with dilute HNO_3, and add a little of the clear solution to some $AmHMoO_4$-solution. Shake and stir the liquid well; and if no precipitate forms, warm *very gently;* a yellow precipitate shows the *Presence of* PO_4.

If AsO_4 has been detected (1052) the above test for PO_4 is only trustworthy when the yellow precipitate has been obtained either in the cold, or by employing a *very gentle* heat. If any doubt is felt concerning the presence of PO_4, boil some of the substance with H_2SO_3 solution (1011), and examine for PO_4 after the AsO_4 has been entirely separated by passing H_2S into the boiling HCl solution. A precipitate which has been obtained with $AmHMoO_4$ may be proved to contain phosphate by dissolving it in AmHO, adding AmCl and $MgSO_4$, filtering, and dropping $AgNO_3$-solution upon the precipitate; if phosphate is present the white precipitate turns *yellow*, if arsenate is present the precipitate becomes *brown.*

1054. **Borate.**—Warm a portion of the substance with a little dilute HCl, dip into the solution a strip of turmeric-paper, and dry the paper in a steam-oven or at a gentle heat. The change of colour of the turmeric to *reddish-brown*, which becomes *blue-black* when the paper is moistened with AmHO, shows the *Presence of* BO_3.

1055. **Fluoride**—Pour upon a portion of the powdered substance, contained in a leaden cup or in a platinum crucible, some strong H_2SO_4. Coat a watch-glass with a film of paraffin wax, and trace some lines through the film with the point of a penknife. Place the watch-glass as a cover upon the metal vessel, and gently warm the vessel for about ten minutes. Then remove the wax. If the lines which were traced are etched upon the glass (606), the *Presence of F is shown.*

If SiO_2 is known to be present (984), the test for fluoride must be made by conducting the gas, which is evolved when the substance is heated with strong H_2SiO_4, into dilute AmHO; a deposit of gelatinous H_2SiO_4 proves the *Presence of F.*

1056. **Sulphide.**—If the presence of sulphide has been indicated by the tests in 992, 997, and 999, or if the presence of a sulphide is suspected and these tests have not indicated its presence, the substance may be boiled with KHO for some time, and alkaline $Pb\bar{A}_2$-solution may be added to the clear liquid; the appearance of a black precipitate or coloration indicates the *Presence of Sulphide.*

SPECIAL PROCESSES OF EXAMINATION.

1057. This concludes the ordinary methods of examining a soluble substance. The remaining paragraphs of this Section treat of the special processes which are sometimes necessary for the analysis of Group III. (1058–1066), and processes for the analysis of Metallic Substances (1067–1073), of insoluble Bodies (1074–1079), of Silicates (1080–1082), and of Cyanogen compounds (1083–1089).

Tables are also appended indicating where the Rarer Elements are detected in an ordinary analysis (1090–1091).

A special scheme for analysing Organic Substances is also inserted (1092–1106).

A list of commonly occurring Minerals, with their chemical formulæ, will be found in paragraph 1172.

REMARKS ON THE PRECIPITATION OF GROUP III.

1058. Separation of Groups III.A. and III.B.—In the General Table (**1007**) it is assumed that, in the absence of phosphate, the two Sub-groups III.A. and III.B. can be separated from one another by adding first AmCl and then excess of AmHO to the solution. It is assumed that Group III.A. alone will be thus precipitated, and that Group III.B. will be afterwards precipitated by adding Am_2S to the filtrate.

It is true that AmCl entirely prevents the precipitation of Group III.B. by AmHO, if certain other metals are absent, and if the solution is kept covered from the air. But the members of Group III.A., if they are present in the solution with those of Group III.B., are precipitated by AmHO; and the presence of AmCl will not prevent Mn and Zn from being partially precipitated with them. Small amounts of Mn and Zn are frequently precipitated in this way, especially with Fe in Group III.A.

Hence if Mn or Zn is present in small quantity only, it may be entirely precipitated in Group III.A. Mn is readily detected in the ordinary examination of this precipitate by Table III.A., since it yields a *green* mass with fused Na_2CO_3 and KNO_3. But if Zn has been entirely precipitated in Group III.A., its presence will certainly not be detected in Table III.A., and it may be altogether overlooked.

1059. The method of dissolving the Group III.A. precipitate in HCl, and reprecipitating it with AmHO, to some extent meets this difficulty, especially if these processes are repeated several times in succession. But when a precipitate is obtained in Group III.A., and traces of Mn, and more particularly of Zn, have to be tested for, it is preferable to precipitate Groups III.A. and III.B. together, by adding AmCl, AmHO, and Am_2S, in succession, and boiling. This precipitate is then examined for Groups III.A. and III.B. by

Table III.c. (1063), or Table III.d. (1064); and the filtrate is examined for Groups IV. and V. according to the directions in the General Table (1007).

It must be understood that, if no precipitate is produced by the addition of AmCl and AmHO, Am_2S may be added at once. Any precipitate, which is formed, is then examined by Table III.b.; since in the absence of Group III.a. the members of Group III.b. are not precipitated by AmHO in the presence of AmCl.

1060. **The Presence of (PO_4) in the HCl solution,** which has to be examined for Groups III., IV., and V., involves no special procedure, if AmHO added after AmCl produces no precipitate; since the phosphates of Groups III. and IV. and of Mg must be absent. If, however, a precipitate is formed on adding AmCl and AmHO, the directions for precipitation, which are given at the head of Table III.p. (1065), should be followed, and the precipitate should be examined by that Table. The method of examination of this precipitate may be simplified by using Table III.p_1 (1066).

1061 **Explanation of the Phosphate Table III.p.**—The reason for the departure from the ordinary course of analysis in the above case is the following. The phosphates of Al, Cr, Ba, Sr, Ca and Mg are completely precipitated by AmHO. The phosphates of Ni, Co, Mn, Zn. and Fe are only partially precipitated by AmHO; the metals are, however, entirely precipitated from these phosphates by Am_2S in the form of sulphides. Hence if the precipitates, obtained by AmHO and by Am_2S respectively, are mixed, the whole of the metals are obtained in the conjoint precipitate.

In obtaining the precipitate for Table III.p., the precipitates produced by AmHO and by Am_2S must be filtered and washed separately; since phosphates of Fe, Zn, Mn, Ni and Co are converted by Am_2S into sulphides, with formation of ammonium phosphate in solution. This soluble phosphate would precipitate Ba, Sr, Ca, Mg, as phosphates, even if they were not originally present in that condition. This precipitation would not only complicate the process of analysis, but would also render it impossible to state whether Ba, Sr, Ca and Mg were originally present in solution as phosphates or not.

The phosphates of Groups III. and IV. and of Mg are accordingly first precipitated by AmCl and AmHO; then any members of Group

III.B. and any remaining traces of phosphates of Group III. are precipitated from the filtrate by Am_2S. The two precipitates are mixed and treated with Am_2S, which will dissolve away the (PO_4) from the phosphates of Fe, Zn, Mn, Ni, Co, leaving the other phosphates undecomposed. Hence, if this precipitate is filtered off and the filtrate is tested with AmCl, AmHO and $MgSO_4$, the formation of a white crystalline precipitate will indicate the presence of (PO_4), and will indirectly establish the presence in the original precipitate of phosphate of some one or more of the metals Fe, Zn, Mn, Ni, Co.

Oxalates, Borates, Fluorides, and Silicates of Ba, Sr, Ca, Mg would likewise be precipitated by AmHO in Group III.A. But the evaporation of the HCl-solution, after passing H_2S in the General Table, causes H_3BO_3 and HF to be volatilised, and H_2SiO_3 to become insoluble. Oxalates are also decomposed by the gentle ignition of the solid substance which remains after evaporation. Hence the above salts will not be precipitated if the solution has been evaporated to dryness, and the residue has been gently ignited before Group III.A. is precipitated.

The Principles on which the Method for Separating and Detecting the Metals, which is drawn out in Table III.P., is founded are —

1. The insolubility of the phosphates of Al, Fe and Cr in $H\bar{A}$ in the presence of an alkaline acetate; the other portions of the precipitate being soluble in the acid.

2. The precipitation of all the (PO_4), which was present in the $H\bar{A}$ solution in combination with Ba, Sr, Ca, or Mg, by the addition of Fe_2Cl_6 (577).

3. The further separation of the phosphates of Al, Fe and Cr is somewhat complicated by the fact that $AlPO_4$ is only decomposed by fusion with alkaline carbonate, when SiO_2 is also present; and this SiO_2 has to be removed after the fusion by methods which will be intelligible on reference to paragraphs 602, 599.

An alternative and simpler method of examining a precipitate of Al, Fe and Cr phosphates, by fusion with KHO and KNO_3, is given in Table III.P_1 (1066).

The Best Course to be Pursued in precipitating and examining Group III. is described in paragraph **1062.**

RULES FOR THE PRECIPITATION AND EXAMINATION OF GROUPS III.A. AND III.B.

1062. The Following Rules may be laid down for precipitating and detecting the members of Groups III.A. and III.B. in the HCl solution, a small portion of which has been tested for (PO_4) by $AmHMoO_4$ (1007). For an explanation of these rules see paragraphs 1058-1061.

I. If the Addition of AmHO after AmCl causes no Precipitate, the absence of Al, Fe, Cr and of their phosphates, as well as of phosphates of Ba, Sr, Ca and Mg, is proved. Am_2S is then at once added, and the liquid is boiled. Any precipitate which forms is examined for the members of Group III.B. by Table III.B. (1028). This course is pursued even if (PO_4) is present, since metals of Groups III. and IV. and of Mg cannot be present as phosphates.

II. If the Addition of AmHO after AmCl causes a Precipitate and (PO_4) is not Present, Al, Fe, Cr must have been in solution. The plan of precipitation will in this case depend upon the object of the analyst.

If small quantities of Zn need not be tested for, the method given in the General Table (1007) may be followed.

If traces of Zn have to be tested for in the solution, add AmCl, then excess of AmHO and of Am_2S; boil, filter, and examine the precipitate by Table III.C. (1063) or III.D. (1064).

The method described in Table III.C. gives trustworthy results, and is to be used for very careful analysis. The method in Table III.D. is, however, much more simple, and is sufficiently accurate for general use.

III. If the Addition of AmHO after AmCl causes a Precipitate, and (PO_4) is Present, the method of precipitation to be adopted, together with the Table for the examination of the Group precipitate, will be found in paragraph 1065.

The shorter method in paragraph 1066 may be employed: but it gives less information as to which metals were present as phosphates in the original solution.

1063. TABLE III.C.—THE SEPARATION OF GROUPS III.A. AND III.B. BY $BaCO_3$.

The precipitate may contain Fe, Al, Cr, Zn, Mn, Ni, Co. Rinse it off the filter into a porcelain dish, using as little water as possible, add some strong HCl and boil, adding at intervals a small crystal of $KClO_3$, until all is dissolved but a small quantity of yellow sulphur. Evaporate very nearly to dryness, dilute with a little water, and pour, through a filter if necessary, into a small flask. Cool, pour in a small quantity of $BaCO_3$ suspended in water, cork the flask tightly and shake well; repeat the addition of $BaCO_3$ and agitation until the precipitate is distinctly whitened by the excess of $BaCO_3$: then shake well, cork the flask, and allow it to stand by for at least fifteen minutes, occasionally shaking it vigorously. Let the precipitate subside, filter; wash the precipitate with a little cold water allowing the washings to run through into the filtrate, then wash thoroughly, rejecting the washing-water:—

<table>
<tr>
<td colspan="2">1. *Precipitate* may contain Fe, Al, Cr, and $BaCO_3$; dissolve it in as little boiling HCl as possible, remove Ba from the *boiling* solution by adding *boiling* dilute H_2SO_4 gradually, until after allowing the precipitate to settle, a few additional drops of acid cause no further precipitate (Note 1, below), filter, add pure NaHO in excess to the filtrate, boil and filter.</td>
<td colspan="3">2 *Filtrate* may contain Zn, Mn, Ni, Co, and $BaCl_2$. Remove Ba by adding to the *boiling* liquid *boiling* dilute H_2SO_4 gradually, until the last few drops produce no further precipitate in the clear liquid from which the $BaSO_4$ has been allowed to settle; filter; add pure NaHO in excess to the *cold* filtrate, stir well and filter:</td>
</tr>
<tr>
<td>*Precipitate:* examine for Fe and Cr by column 2, Table III.A (1027).</td>
<td>*Filtrate:* examine for Al by Column 1, Table III A (1027).</td>
<td colspan="2">*Precipitate* may contain Mn, Ni, Co, rinse it off the filter into a porcelain dish with as little water as possible, add some strong HCl and boil; evaporate nearly to dryness, add a little strong solution of $Na\bar{A}$ to the liquid, pass H_2S to saturation, filter.</td>
<td rowspan="2">*Filtrate* pass H_2S a white precipitate indicates *Presence of Zn*.</td>
</tr>
<tr>
<td colspan="2">*Note* 1.—The separation of Ba may be neglected here, the precipitate being at once boiled with NaHO: in this case much white $BaCO_3$ will remain with the Fe_2O_3 after fusion to separate Cr; it will however dissolve with the Fe and will cause no complication.</td>
<td>*Precipitate:* examine for Ni and Co according to 1029.</td>
<td>*Filtrate:* add AmHO in excess; a flesh coloured precipitate shows: *Presence of Mn*.</td>
</tr>
</table>

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1064. TABLE III.D.—FOR EXAMINATION OF

Remove the precipitate produced by AmCl, AmHO, and Am_2S pouring in dilute HCl and gently stirring and shaking the liquid: filter (see Note 1):—

<table>
<tr><td rowspan="3"><i>Residue</i> if black may contain NiS and CoS; examine it as directed in 1029.</td><td colspan="2"><i>Filtrate</i> may contain Al, Fe, Cr, Zn, Mn Carefully note its of this observation, examine the liquid by column I. or II.</td></tr>
<tr><td colspan="2">1 <i>The liquid is perfectly colourless Absence of Cr.</i>
Boil the liquid in the dish for a few minutes until it no longer smells of H_2S, then add a small crystal $KClO_3$ and boil down to a small bulk; cool, add NaHO in excess, stir well and filter:</td></tr>
<tr><td><i>Precipitate</i> may contain Fe, Mn. Dry and fuse it on platinum foil with Na_2CO_3 and KNO_3, a blue green mass shows:
<i>Presence of Mn</i>.
Boil the foil in a porcelain dish with water for some time, and if any undissolved residue is left, decant the liquid, boil the residue with HCl, and add KCyS; a blood red coloration shows:
<i>Presence of Fe</i> (Note 4, 1027).</td><td><i>Filtrate</i> may contain Zn, Al. Divide into two equal parts (Note 2)
Into one portion pass H_2S; a white precipitate forms at once
<i>Presence of Zn</i>.
To the other portion add AmCl in excess, and heat; a colourless flocculent precipitate
<i>Presence of Al.</i></td></tr>
</table>

Note 1.—A mere milkiness, due to the separation of sulphur, shows the

Note 2.—Al and Zn may also be detected without dividing the filtrate by
colourless flocculent precipitate shows *Presence of Al:* this is filtered off and H_2S

GROUPS III.A. AND III.B., WHEN MIXED.

from the filter by opening the filter out inside a porcelain dish, take out the filter paper, stir well, and if any black residue is left,

<table>
<tr><td colspan="3">colour, which is best seen by pouring it into a white porcelain dish, and according to the result</td></tr>
<tr><td colspan="3">II. <i>The liquid has a violet or bright green colour: Presence of Cr.</i>
Boil the liquid in the dish until H_2S is no longer smelt, drop in a small crystal of $KClO_3$ and boil down nearly to dryness, dilute with a little water, pour into a small flask and add $BaCO_3$ suspended in water gradually whilst constantly shaking the liquid until the excess of $BaCO_3$ whitens the precipitate, cork the flask and allow it to stand for not less than fifteen minutes, occasionally shaking it well; filter, wash first with cold water letting the washings run into the filtrate, then with boiling water rejecting the washings:</td></tr>
<tr><td rowspan="2"><i>Precipitate</i> may contain Fe, Al, Cr; examine it by column 1, Table III C (1063)</td><td colspan="2"><i>Filtrate</i> may contain Zn, Mn, boil, and whilst boiling add <i>boiling</i> dilute H_2SO_4 gradually until the last few drops cause no further precipitate, filter from $BaSO_4$; cool, add pure NaHO in excess, stir well and filter:</td></tr>
<tr><td><i>Precipitate:</i> dry and fuse with Na_2CO_3 and KNO_3 on platinum foil, blue green mass
<i>Presence of Mn.</i></td><td><i>Filtrate:</i> pass H_2S; white precipitate.
<i>Presence of Zn.</i></td></tr>
</table>

absence of Ni and Co, and does not render filtration necessary.
adding to it HCl gradually until it becomes acid, then AmHO until alkaline, e
is passed into the filtrate, a white precipitate shows *Presence of Zn*.

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1065. TABLE III.P.—FOR EXAMINATION

If (PO_4) is found in the HCl solution in the General Table
on addition of AmCl and excess of AmHO, the liquid containing the
washed well with hot water. To the filtrate Am_2S is added in excess,
for Groups IV. and V. (1007, 1020). The two precipitates yielded by
well with a little Am_2S, then filtered, and the residue on the filter
adding $MgSO_4$; if this is present, Fe, Zn, Mn, Ni, Co, one or more,

The residue left after stirring with Am_2S may contain Ba, Sr,
Cr as hydrate or phosphate. Remove it from the filter and heat
$KClO_3$ occasionally, until solution is complete. Then either evaporate
sufficient quantity to just redissolve the precipitate. Filter off S if
as long as any precipitate is produced. Heat gently, and filter while

<table>
<tr><td colspan="4"><i>Filtrate:</i> Add Fe_2Cl_6 (Note 2) drop by drop as long as a precipitate forms, and until the liquid, after being well stirred or shaken, remains reddish addition of Fe_2Cl_6 in large excess must be carefully avoided. Warm gently for some time, filter whilst hot, and wash with hot water:</td></tr>
<tr><td colspan="3"><i>Filtrate</i> Add AmCl, AmHO in excess (Note 3), and Am_2S filter</td><td rowspan="3"><i>Precipitate</i> containing $FePO_4$ may be rejected.</td></tr>
<tr><td colspan="2"><i>Filtrate:</i> Add Am_2CO_3: filter:</td><td rowspan="2"><i>Precipitate;</i> examine by Table III.C. or III D (1063, 1064) for Zn, Mn, Ni, Co, also for Al and Cr
Test a portion of the original solution, or the solution of the substance in HCl, for Fe'' and Fe''' according to Note 4 (1027).</td></tr>
<tr><td><i>Precipitate</i> may contain $BaCO_3$, $SrCO_3$, $CaCO_3$ Examine this precipitate by Table IV. (1031–1034)
Ba, Sr, Ca, if found in this precipitate, were present in the solution as phosphates</td><td><i>Filtrate</i> may contain Mg, after removing any traces of Ba, Sr, Ca (Table 1035, Col I) add Na_2HPO_4, warm and shake well, white crystalline precipitate:
<i>Presence of Mg as phosphate</i></td></tr>
</table>

Note 1.—An insoluble residue here may contain $SrSO_4$ and $BaSO_4$, and must
sent originally as phosphate.

Note 2.—A few drops of Fe_2Cl_6 may be added to a small part only of the
be examined at once for Groups III. and IV. and for Mg, without adding Fe_2Cl_6.

Note 3.—Since this filtrate has to be tested for Groups III.A. and III.B., it
analysing the precipitate by Table III.C., or by precipitating Groups IIIA.
precipitates by Tables III.A. and III.B. (1027, 1028).

Note 4.—Since $CrPO_4$ is rarely present, this precipitate may generally be tested
may be detected by adding AmCl in excess, which gives a gelatinous precipitate;
and adding KCyS. An alternative method of examining this precipitate by

OF PHOSPHATES IN GROUP III.

(**1007**) after precipitating Groups I. and II., and a precipitate forms
precipitate is gently heated, filtered quickly, and the precipitate is
and the liquid is boiled; it is then filtered, and the filtrate is examined
AmHO and by Am_2S are transferred to a porcelain dish and stirred
is washed well. The filtrate should be examined for (PO_4) by
must have been present as phosphate.

Ca, Mg as phosphate; Fe, Zn, Mn, Ni, Co as sulphide; and Al,
it with a little dilute HCl in a porcelain dish, dropping in a crystal of
very nearly to dryness, or add AmHO in slight excess and then HCl in
necessary (*Note* 1), and add a solution of $H\bar{A}$ and $Na\bar{A}$ (**1162**, 84),
hot:—

<table>
<tr><td colspan="4"><i>Precipitate</i> may consist of $FePO_4$ $AlPO_4$, $CrPO_4$ (Note 4) Dry the precipitate on the filter, and fuse it for a short time in a platinum crucible or on platinum foil with a mixture of finely powdered SiO_2, Na_2CO_3, and KNO_3. When cold dissolve by boiling with a little distilled water, add Am_2CO_3, allow to stand for a short time, stirring occasionally, let the precipitate subside, filter:</td></tr>
<tr><td rowspan="3"><i>Filtrate</i>: add $H\bar{A}$ until the solution is acid, boil for a short time, then add $Pb\bar{A}_2$, yellow precipitate of $PbCrO_4$
<i>Presence of Cr, as phosphate.</i>
<i>Note.</i>—A white precipitate on addition of $Pb\bar{A}_2$ may be disregarded</td><td colspan="3"><i>Precipitate</i> may contain H_4SiO_4, Fe and Al as silicates, and $Fe_2(HO)_6$ Acidify with HCl, evaporate to dryness, and heat the dry residue gently Warm with a few drops of strong HCl, add hot water, and filter:</td></tr>
<tr><td colspan="2"><i>Filtrate</i>. add pure NaHO in excess, boil and filter:</td><td rowspan="2"><i>Residue</i> is SiO_2 and may be neglected.</td></tr>
<tr><td><i>Filtrate</i>: add AmCl in excess, boil. white, gelatinous precipitate:
<i>Presence of Al as phosphate.</i></td><td><i>Precipitate</i> is brown $Fe_2(HO)_6$: dissolve by boiling with HCl, and add KCyS: blood-red coloration:
<i>Presence of Fe as phosphate.</i></td></tr>
</table>

be examined by the latter part of 1017: Ba or Sr, if found here, was pre-

filtrate and the liquid heated; if no precipitate is produced the other portion may

may be examined either by adding AmCl, AmHO, and Am_2S together, and
and III.B. separately by AmCl and AmHO and by Am_2S, and examining the

by boiling it with excess of NaHO, and filtering; $AlPO_4$ will be in solution, and
$Fe_2(HO)_6$ will remain as a precipitate, and may be detected by dissolving in HCl
fusion with KHO and KNO_3 is given in Table III.P_1 (1066).

1066. TABLE III.P$_1$.—SECOND PHOSPHATE TABLE.

Dissolve the Precipitate, which has been produced by the addition of AmHO in excess after AmCl (1007), in the least possible quantity of HCl: add to this solution a few drops of HĀ and a considerable quantity of AmĀ-solution (1161, 54): then add $FeCl_3$ drop by drop, and with constant stirring, until the liquid just becomes permanently red in colour.

If the liquid contains no precipitate after it has been mixed with a few drops of $FeCl_3$, add a small quantity of Am_2HPO_4 or Na_2HPO_4 (see *Note*, page 397): and then stir with $FeCl_3$, added gradually drop by drop until the liquid is permanently reddened.

Now boil the turbid liquid for several minutes, and filter it while it is still at boiling heat (*Note*, 1144):—

<table>
<tr>
<td colspan="2">The Precipitate may be neglected, if the addition of AmĀ has caused no precipitate, but a precipitate has been produced by the subsequent addition of $FeCl_3$, since in this case metals of Group III.A must be absent.
If, however, the addition of AmĀ has caused a precipitate, the above precipitate which has been separated from the boiling liquid must be examined as follows —
Dry the precipitate on the filter; then remove the dried particles, and heat them with a fused mixture of KHO and KNO_3 on platinum foil. Allow the fused mass to cool, boil it with water, and filter the solution :—</td>
<td rowspan="2">The Filtrate should be free from phosphate, and should give no precipitate when excess of AmHO is added after AmCl.
Examine this filtrate for Groups III B, IV, V, according to the directions given in the General Table (1007).</td>
</tr>
<tr>
<td>The Residue will consist of Fe_2O_3 and may be neglected, since $FeCl_3$ has been added as a reagent.
But the HCl solution of the original substance must be tested for Fe'' and Fe''' by adding to separate portions of it K_3FeCy_6 and KCyS respectively (1027, Note 4)</td>
<td>The Filtrate will be yellow or colourless.
If it is yellow, Cr is present, confirm its presence by acidifying a portion of the liquid with HA and adding PbA_2: a yellow precipitate shows the Presence of Cr.
Add to the colourless filtrate, or to the remainder of the yellow filtrate, AmCl in large quantity: a colourless gelatinous precipitate shows the <i>Presence of Al.</i></td>
</tr>
</table>

Note.—The addition of $Am\bar{A}$ precipitates $FePO_4$, $AlPO_4$ and $CrPO_4$ only partially if PO_4 is present in small quantity only, and the liquid is subsequently not entirely freed from PO_4 by being boiled. The addition of the soluble phosphate renders the precipitation of $FePO_4$ and $AlPO_4$ complete; while the excess of iron, together with any dissolved $CrPO_4$, is completely precipitated by the subsequent boiling of the liquid.

ANALYSIS OF METALS AND ALLOYS.

1067. The metal is first reduced to powder, filings, or thin turnings or shavings. The following Preliminary Examination (1068) is then made, and one of the methods described in paragraphs 1069, 1070 is adopted for the full examination.

The First Method should be employed for the examination of a metal or alloy of unknown composition; since it enables all metals to be detected, even when they are present in small quantity only.

The Second Method leaves Sn, Sb, Au and Pt undissolved, while other metals pass into solution.

This method is not recommended as a *general one*, since the following complications may occur.

If As is present with Sn, a part or the whole of the As may remain in the residue. If Pt is present with a sufficient quantity of Ag, the Pt may pass entirely into solution. Sb will always partly enter into solution.

The process, however, is useful in cases where an alloy, which is known to contain a large proportion of Sn or Sb, has to be examined for other metals: since the other metals are dissolved away at once from the bulk of the Sn or Sb, and a solution is obtained which is better suited for general analysis.

PRELIMINARY EXAMINATION.

1068. Note the General Appearance and Properties of the Substance, its colour, any characteristic smell which may be given off when it is rubbed with the hand, also whether it is crystalline or not; and whether it is attracted by a magnet (Fe, Ni, Co, &c.).

Note the hardness of the metal, by seeing if it can be scratched or cut by a steel knife; also whether it breaks to powder (brittle), or flattens out (malleable), when it is struck smartly with a hammer. Then try the following experiments with separate small portions of the substance:—

I. Heat with Na_2CO_3 on charcoal in the inner blowpipe-flame (991, 3, and 992).

II. Fuse into a colourless borax-bead in the outer blowpipe-flame (991, 2).

III. Try the flame-coloration (990).

IV. Heat in an ignition tube, and note whether a metallic sublimate forms of Cd, As, or of globules of Hg; or whether S sublimes from a sulphide.

V. Combined P, As, S, and Si can frequently only be detected, after they have been converted into phosphate, arsenate, sulphate, and silicate, by dissolving the substance in strong HNO_3 or aqua regia, or by fusing it with KNO_3 and Na_2CO_3 and then dissolving in water and acidifying with HCl. the solution is then tested by paragraphs 1053, 1052, 1048, and by 1017 after evaporation to dryness.

VI. C from metallic carbide may remain, when the metal is dissolved by HCl or by $CuCl_2$-solution, as a black residue, which can be burnt away on platinum foil at a bright red heat (Exp. 81, 105). Or the carbon may escape, during solution of the metal in acid, as a hydrocarbon, which produces H_2O and CO_2 by its combustion.

SOLUTION AND EXAMINATION OF A METAL OR ALLOY.

1069. METHOD I.—Pour some rather dilute HCl upon the powdered metal in a small flask with a glass funnel in its neck (fig. 38, p. 61), and heat for some time just short of boiling. If the metal dissolves readily, continue to heat until the metal is completely dissolved, and examine the HCl solution according to the General Table (**1007**).

Frequently HCl alone does not effect complete solution. Two or three drops of strong HNO_3 should then be poured in, and more HNO_3 and HCl should be added occasionally when the action ceases or when red fumes are no longer given off on heating. When the metal has entirely disappeared, add a little more strong HCl and boil as long as any Cl or reddish fumes are given off; then dilute with a little water, heat to boiling, and cool; filter, if there is any white residue :—

Residue, if crystalline, is probably $PbCl_2$, which will dissolve entirely if it is washed with sufficient boiling water. In this solution the presence of Pb is confirmed by adding K_2CrO_4, which gives a yellow precipitate soluble in excess of KHO, showing *Presence of Pb.* If any residue is left after washing well with boiling water, it is probably AgCl; confirm the presence of Ag by pouring upon the residue hot AmHO, it dissolves completely and is reprecipitated on adding HNO_3 in excess :— *Presence of Ag.*	*Filtrate:* dilute largely with cold water (*Note*), and whether this causes a precipitate or not, pass H_2S to saturation into the cold solution; examine any precipitate thus produced by Table II. (**1024**), and proceed to examine the filtrate for Groups III., IV., and V., as directed in the General Table (**1007**). *Note.*—A white precipitate, appearing on dilution, is due to the presence of Bi, Sb, or Sn.

1070. METHOD II.—Pour upon the finely-divided metal some strong HNO_3,* and heat in a small flask with a watch-glass or funnel on its neck (fig. 38, p. 61), as long as any red fumes appear: one of two results will occur :—

<table>
<tr><th>I.</th><th colspan="2">II.</th></tr>
<tr><td rowspan="2"><i>The Substance dissolves completely, with or without addition of water</i> Absence of Pt, Au, Sb, Sn (Note 1)
Examine the solution, after boiling nearly to dryness and diluting with water (Note 2), by the General Table (1007)</td><td colspan="2"><i>A Residue is left:</i> add some hot water and boil, then filter, and wash the residue on the filter well with boiling water (Note 2). Examine the solution by I. The residue will consist either of :—</td></tr>
<tr><td>1 <i>An Entirely Metallic or Black Powder</i>, probably Pt or Au Dissolve this residue and examine the solution as is directed in paragraph 1073.</td><td>2 <i>Or of a White Powder</i> containing Sn, Sb, As, possibly also Pt and Au concealed in it (Note 3).</td></tr>
</table>

Note 1.—The solution is liable to contain small quantities of these metals, which must always be tested for in a careful analysis.

---

* If Hg has been found in the Preliminary Examination and small quantities of Ag have to be tested for, the Hg should be expelled by heating the substance strongly in a porcelain crucible before it is dissolved in acid, since $Hg(NO_3)_2$ tends to prevent the precipitation of Ag by HCl in Group I.

Note 2.—BiOCl will often be precipitated by dilution, but the precipitate will disappear on adding HCl and boiling, and is thus easily distinguished from the Group I. precipitate.

Note 3.—If much residue is obtained, a small quantity of it may be heated in a test-tube with HCl, adding $KClO_3$ · if it dissolves entirely, dissolve the whole of the residue, and examine the solution by Table II., commencing at paragraph 1026 if it refuses to dissolve, proceed with the rest of the residue as is directed below. If the residue is small in quantity examine it at once as is directed below.

1071. Examination of the Non-metallic Residue Insoluble in HNO_3.—Dry the residue on the filter at a gentle heat, mix it thoroughly with about an equal quantity of powdered Na_2CO_3 and $NaNO_3$, and add the mixture gradually to some fused $NaNO_3$ contained in a porcelain crucible. Then pour the melted substance out into a porcelain dish; allow it to cool, pour upon it cold water and let it stand for some time. Then crush the mass with a pestle, and stir it occasionally; filter, and wash the residue on the filter with dilute alcohol, throwing away the washings :—

Residue may contain Sn, Sb, Pt, Au. Place it in a small porcelain dish, pour in a little HCl and heat, then add water; whether the residue has dissolved or not, place in the liquid a strip of Pt-foil and drop upon it a piece of pure Zn; remove the platinum strip in a few seconds, if it is stained black *Sb is present.* Wait until the evolution of H ceases, taking care that there is some Zn left undissolved, and adding more Zn if the first piece has entirely dissolved. The residue in the dish may consist of Sn, Au, and Pt; remove the Zn, rinsing off any substance, adhering to it, into the dish; stir the liquid in the dish well, then pour off the liquid, carefully leaving the residue; pour in water, stir well, and again pour off carefully. Boil the residue for some time with strong HCl in a test-tube, dilute, decant, add $HgCl_2$, a white precipitate forms :—*Presence of Sn.* ***Residue:*** dissolve by warming with HCl and HNO_3, and examine the solution for Au and Pt by (1073), using only the left hand portion of the Table.	***Filtrate*** may contain (AsO_4): add HNO_3 until the solution is acid and boil, evaporating the liquid in a dish if very bulky. Pour into half this solution $AgNO_3$ as long as it gives any precipitate, and add gradually AmHO diluted with 10 or 12 times its bulk of water, a brown precipitate shows — *Presence of As.* To the other half of the acid solution add AmHO in excess, then $MgSO_4$, and rub the inside of the vessel with a glass rod; a white crystalline precipitate, often appearing only after some time, shows :— *Presence of As.*

1072. Detection of Au and Pt.—The Residue may contain Au, Pt, (Sn, Sb). Remove any fragments of Zn, boil the residue with strong HCl, allow to settle, and decant :—

<table>
<tr>
<td colspan="2">1073. <i>Residue</i>*; pour upon the residue in the dish a little HCl, add several drops of HNO_3, and boil gently very nearly to dryness , add some KCl solution and evaporate once more very nearly to dryness. Pour some absolute alcohol into the cool dish and stir well for a time, allow the precipitate to settle and decant the liquid, wash the precipitate by stirring it with a little more alcohol and decant the liquid when the precipitate has settled —</td>
<td rowspan="2"><i>Solution</i>; to one part add $HgCl_2$ a white precipitate — <i>Presence of Sn.</i>
In the other part of this solution immerse a strip of Pt and drop a piece of Zn on the Pt , a black stain on the Pt shows ,— <i>Presence of Sb.</i></td>
</tr>
<tr>
<td><i>Precipitate</i> will consist of yellow K_2PtCl_6 dissolve it in a little boiling water, add several drops of HCl, then $SnCl_2$ an orange red coloration confirms the <i>Presence of Pt</i></td>
<td><i>Solution</i> will be yellow if Au is present evaporate on a water-bath, dissolve in a little water and add a few drops of freshly prepared $FeSO_4$ solution a blue liquid :— <i>Presence of Au</i></td>
</tr>
</table>

* If Sb has not been detected already, this residue should be examined for Sb. Boil it once more for some time with strong HCl to remove all Sn, wash the residue well by decantation and boil it in the dish with $H_2\overline{T}$, adding a few drops of HNO_3, decant, and test the liquid for Sb by adding HCl and passing H_2S : the residue is then examined as is directed above (1073).

EXAMINATION OF SUBSTANCES INSOLUBLE IN WATER AND ACIDS.

1074. An insoluble Substance may Consist of one or more of the following substances, all of which are white except Fe_2O_3, Cr_2O_3, $FeCr_2O_4$, $PbCrO_4$, S, C, native SnO_2 and CaF_2, and AgCl which has been exposed to light.

In the following list those substances which are embraced in round brackets ( ) may *possibly* be present, those in square brackets [ ] *improbably*, because they are soluble in much water.

1. $BaSO_4$	Insoluble in water and acids.
2. $SrSO_4$	Insoluble ,, ,,
3. $[CaSO_4]$ 4. $(PbSO_4)$	Not perfectly insoluble in water, soluble in hot HCl, and should therefore pass into the acid solution.
5. $PbCrO_4$	Insoluble after being strongly heated.
6. $[PbCl_2]$	Soluble in boiling water, and should therefore have been removed if the residue was well washed with boiling water.
7. $AgCl$	This may have been originally present as such, or may have been derived from the use of HCl in making the solution, or by the action of aqua regia on the insoluble substances AgBr, AgI, AgCy, Ag_3FeCy_6, Ag_4FeCy_6.
8. SiO_2	Either uncombined, or as a silicate.
9. (Al_2O_3) 10. (Fe_2O_3) 11. (Cr_2O_3)	Insoluble after being strongly ignited, but usually dissolved by long boiling with strong HCl.
12. $FeCr_2O_4$	Chrome iron ore, native.
13. (SnO_2)	Native or ignited.
14. Sb_2O_5, Sb_2O_4	
15. CaF_2	Also a few other fluorides, and some metaphosphates and arsenates.
16. S	Yellow, slowly soluble in strong HNO_3, giving red fumes, and yielding H_2SO_4.
17. C	Black, and quite insoluble.

If sufficient of the substance is available, the Preliminary Examination (1075) may be made on a portion of it. In case the quantity of substance is small, however, the whole of the substance must be employed for the examination by fusion (1076).

Preliminary Examination of Insoluble Substances.

1075. The substance must be in the state of dry powder. Make a careful examination of it with a pocket lens. **Expts. I. and II. need only be made if the substance is light in colour.**

Experiment.	Observation.	Inference.
I Observe whether the substance darkens when allowed to stand in the light for some time.	**The colour changes to violet or black**	**Presence of AgCl.**
II. Pour a little Am_2S upon a portion of the substance on a watch glass.	The substance blackens; pass on to III. The substance does not blacken; pass on to V., omitting III. and IV., since Pb and Ag must be absent.	Presence of Pb or Ag.
III. Heat some of the substance with a little water and a small piece of KCy; filter off, keeping the residue; to the filtrate add Am_2S.	**A brownish precipitate.**	Presence of AgCl. *Confm.* On warming some of the substance with AmHO, filtering, and adding excess of HNO_3 to the filtrate, a white precipitate forms, which, when shaken well or heated, coagulates into flocks.
IV. Wash the residue from Exp. III. well upon the filter:—		
a. *It is white;* drop Am_2S upon it.	**The residue blackens.**	Presence of $PbSO_4$ or $PbCl_2$.
b. *It is dark coloured:* add water and $H_2\bar{T}$, and boil after adding AmHO in excess; filter, to the filtrate add $H\bar{A}$ in excess and K_2CrO_4.	A yellow precipitate, soluble in KHO.	Presence of $PbSO_4$ or $PbCl_2$.
V. Take up some of the substance upon a moistened loop of platinum wire; heat it for a short time in the inner blowpipe flame, then moisten with a drop of strong HCl, and hold in the outer part of a Bunsen flame.	A *reddish yellow* coloration, *dusky green* when seen through the indigo prism. A *crimson red* coloration appearing *deep red* through the indigo prism. A *yellowish green* coloration. **These colorations may often be seen in succession and further distinguished by the spectroscope.**	**Presence of Ca** **Presence of Sr** **Presence of Ba** **as sulphate.**
VI. Heat in a small test-tube or ignition-tube, then strongly on a piece of porcelain or broken glass, or on platinum foil.	A yellow sublimate forms on the sides of tube. When strongly heated the substance smoulders, and ultimately burns away.	**Presence of S.** **Presence of C.**

Experiment.	Observation.	Inference.
VII. Heat with strong H_2SO_4 in a platinum crucible or leaden cup covered with a watch glass (606) or if a silicate is present examine by passing the gas into AmHO (607)	The glass is etched. A gelatinous precipitate is obtained in the AmHO	Presence of F Presence of F.
VIII Fuse some of the substance in a bead of $NaAmHPO_4$, first in the outer then in the inner blowpipe flame.	Particles are seen floating undissolved in the melted bead	Presence of SiO_2.
	Green coloured bead.	Presence of Cr.
	Reddish brown bead, colourless when cold, and becoming *greenish* in the inner flame	Presence of Fe.

EXAMINATION OF INSOLUBLE SUBSTANCES.

1076. Treatment of the Substance with fused Alkaline carbonate (106, Exp. 84).—Free the substance, if necessary (1075, VI.), from free S or C by igniting it strongly in an open porcelain crucible. Mix the finely-powdered substance with five or six times its volume of *fusion-mixture*, NaC_2O_3 + K_2CO_3, and heat the mixture in a small covered porcelain crucible over the Bunsen-flame (fig. 80) until it melts. It will sometimes be necessary to employ the blowpipe-flame in order to fuse the mass. Keep the mixture in the fused condition for at least ten minutes, and then allow the crucible to cool.

A Platinum Crucible is preferable to a porcelain crucible for this process, since, after the mixture has been fused in porcelain, small quantities of Al_2O_3 and SiO_2 will always be introduced, owing to the action of the alkaline carbonates upon the glaze of the porcelain

If Pb and Ag have been proved to be absent by the Preliminary tests (1075, II.), or if they have been removed by boiling the substance first with KCy-solution, and then with $H_2\overline{T}$ and excess of AmHO and washing well, a platinum crucible may be used.

The Form of Pipe-clay triangle, which is shown in fig. 81, is recommended for supporting the crucible, since the prominences on its sides enable the flame to reach the sides of the crucible.

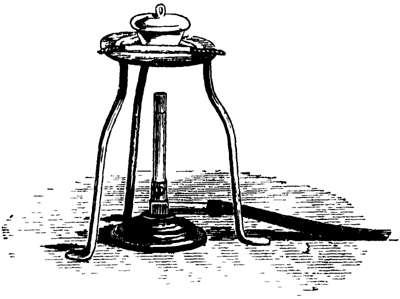
A Gas Blowpipe will be found convenient for fusing the mixture :

see fig. 10 (7), figs. 82, 83 (1118); the petroleum or spirit blowpipe shown in fig. 85 (1119) also serves the purpose well.

Now pour some water into the crucible, and either allow it to stand, or boil the liquid, until the solid mass is loosened from the crucible. Boil this mass in a porcelain dish with distilled water, crushing it by pressure with a pestle if it does not quickly fall to pieces.

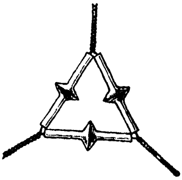
Allow the residue to settle, pour off the solution through a filter, and boil the residue with a little more water; pour off through the same filter, adding this filtrate to the former one.

FIG. 80.



TREATMENT WITH FUSED ALKALINE CARBONATES.

FIG. 81.



PIPE-CLAY TRIANGLE.

The examination of the undissolved residue is described in paragraph **1077**, and that of the filtrate in paragraph **1078**.

1077. Insoluble Residue on the Filter.—Wash the residue well with boiling water, then make a hole in the bottom of the filter, and rinse the residue through into a test-tube by pouring upon it a little boiling dilute HCl (*Note* 1). The residue will dissolve entirely when it is heated with the acid, if the fusion has been continued sufficiently long (*Note* 2). Filter, if necessary, and examine the solution by the General Table (**1007**), bearing in mind that only the metals in paragraph **1074** are likely to be present (*Note* 3).

Note 1.—**If Ag and Pb** are known to be present by the Preliminary Examination (1075, II.), dilute HNO_3 must be used, instead of HCl, for dissolving the residue.

If effervescence is produced by the action of the acid upon the residue, the presence of Ba, Sr, Ca, or Mg is proved.

Note 2.—**A Residue Insoluble in Acid** should be finely powdered and then treated once more with fresh fusion-mixture (1076). If it consists of chrome iron ore, it may be dissolved by heating it with HNO_3 and $KClO_3$.

Note 3.—**The Solution should be Evaporated quite to Dryness** before Group III. is precipitated, and a water-bath should be used towards the end of the evaporation if the substance spirts.

The use of a water-bath is usually necessary if SiO_2 is present in the substance, since silicic acid may pass into the solution, and if it is not completely separated by the process of evaporation (599), its presence may give rise to complications in the analysis.

1078. Aqueous Solution of the Fused Mass—Divide this solution into two parts, (a) and (b).

(a) Add HCl to one part until the liquid is distinctly acid, and evaporate in a porcelain dish to dryness, finishing the process on a water-bath, if necessary, to avoid spirting. Then continue to heat the dish gently over the flame or upon a sand-bath until the residue is quite dry. Pour in a little strong HCl and warm, dilute and heat again; an insoluble residue shows:—**Presence of SiO_2.**

Filter off SiO_2 if necessary, and examine the filtrate by the General Table (1007). Al may be found here, but other metals, such as Cr, Mn, Zn, Sn, Sb, Cu, As, should also be tested for, since their oxides are soluble in alkalis, and may therefore pass into this solution.

(b) Test separate portions of the other part of the aqueous solution for the following acid-radicles as is described below. Unless the fusion-mixture employed was free from chloride, sulphate and phosphate, the tests for these acid-radicles may be of little value.

Chloride: acidify with HNO_3 and add $AgNO_3$; white precipitate, easily soluble in AmHO.

Sulphate: acidify with HCl and add $BaCl_2$; white precipitate insoluble on boiling.

Chromate: acidify with $H\bar{A}$ and add $Pb\bar{A}_2$; yellow precipitate A chromate is also seen by the yellow colour of the solution. Cr thus detected may have been present acting as a metal or as a constituent of the chromate acid-radicle (*Note* 3, 1027).

Phosphate: acidify with HNO_3, add a few drops to some AmHMoO solution, and warm gently; a yellow precipitate forms, often only after a time or when the liquid is gently heated.

If As has been found when H_2S was passed into the hot HCl solution, the presence of (PO_4) must be confirmed in a portion of the filtrate from which the As has been completely precipitated (1053), else the above yellow precipitate may have been due to (AsO_4).

Fluoride: add HCl in excess to a part of the solution, stir well, and let stand until the CO_2 has escaped; then add AmHO in excess, then $CaCl_2$ as long as it produces any precipitate, and let stand for a time.

Filter off and dry any precipitate which forms, and pour strong H_2SO_4 upon it in a platinum crucible covered with a suitably prepared watch-glass (606).

1079. Examination for Na and K.—Since Na and K have been introduced into the substance by the fusion mixture, these metals cannot be tested for in the aqueous solution obtained after fusion.

Unless the analyst is satisfied that the spectroscopic test, made with the special precautions mentioned in paragraph 117, is conclusive, he should proceed to examine the substance for Na and K by paragraph 1081.

The simpler method of examination by paragraph 1082 may be employed if the substance is a silicate: and if K and Na are present in a condition in which they are insoluble in acids, they will usually be contained in a complex silicate.

ANALYSIS OF SILICATES.

1080. The Presence of Silica in a substance will have been shown by the special test (984) or by VIII. in paragraph 1075.

When silica has been found it becomes necessary to examine the substance for all metals, since many silicates, which are soluble when they are alone, become insoluble when they are mixed or combined with other insoluble silicates.

Many silicates may be entirely decomposed by heating them for some time with strong HCl just short of boiling. If the decomposition has been complete, only a colourless residue of silicic acid will remain. This may be identified by its insolubility in fused microcosmic salt (600), and by causing effervescence when it is fused into a bead of Na_2CO_3 (601).

If it is found that the silicate is not completely decomposed by hot strong HCl, it should be treated with fused alkaline carbonates, as is directed in paragraphs 1076, *et seq.* In the examination which follows the fusion, it must be remembered, however, that all metals may be present. The metals Na and K must be tested for specially by paragraph 1081 or 1082.

EXAMINATION FOR NA AND K IN AN INSOLUBLE SUBSTANCE.

Since Na and K cannot be tested for in the solution which is obtained after fusion with alkaline carbonates (1076), a separate portion of the original substance must be examined for these metals by one of the two following processes. The materials which are used in the tests must be perfectly free from K and Na.

1081. Decomposition by means of CaO.—The finely-powdered substance is mixed with its own weight of sublimed and crystallised NH_4Cl in powder, and with eight times its weight of pure $CaCO_3$ (see next page). The mixture is gently heated in a platinum crucible for a few minutes, and is finally kept at a bright red heat for twenty or thirty minutes. The mass will not fuse as a whole, but sufficient CaO will be dissolved by the fused $CaCl_2$, and thus be brought into contact with the silicates, to secure their decomposition.

The cool substance is turned out of the crucible if possible, and is boiled with water for some time after it has crumbled by the slaking of the CaO. The liquid is then filtered, and Am_2CO_3-solution is added to the filtrate until it causes no further precipitate of $CaCO_3$. The filtrate from this precipitate is evaporated considerably, and is then freed from traces of Ca by the addition of $Am_2C_2O_4$. The clear solution will now contain chlorides of K, Na, Li, if these metals were present in the original substance. They may be tested for by the right-hand side of Table V. (1035), and by the spectroscope (113).

The Pure $CaCO_3$, which is required for the above process, is prepared by dissolving marble in HCl, and adding powdered marble in excess and warming. The solution is then mixed with lime-water or milk of lime until it is alkaline in reaction: magnesium, calcium phosphate, and iron are thus precipitated. This solution is heated to about 75° C., and warm Am_2CO_3-solution is added until it causes no further precipitate. The precipitated $CaCO_3$ is filtered off, and is washed well on the filter.

1082. Decomposition by means of HF.—The finely-powdered substance is evaporated several times in a platinum dish or crucible either with hydrofluoric acid and subsequently with strong H_2SO_4, or with five times its weight of finely-powdered calcium fluoride mixed into a paste with strong H_2SO_4.

In either case the mass is finally heated until no more white fumes are evolved. The cool residue is then boiled with water; $BaCl_2$-solution is added as long as it causes any precipitate; then AmHO is added in excess, and Am_2CO_3 solution is poured in as long as it causes any precipitate.

The precipitate is filtered off, and the filtrate is examined for K and Na by Table V. (1035), and by the spectroscope (113).

ANALYSIS OF SUBSTANCES CONTAINING CYANOGEN.

1083. If the substance to be analysed is found to contain cyanogen (983), the usual course of analysis must frequently be somewhat modified, since the presence of cyanogen might otherwise produce confusing results.

The cyanogen may be present as cyanide, sulphocyanide, ferrocyanide, ferricyanide, cobalticyanide, and rarely as a manganocyanide or chromicyanide. It is necessary first to ascertain in what form the cyanogen occurs, by trying the following preliminary experiments on a small portion of the substance. The subsequent procedure is explained in paragraphs 1085–1089.

PRELIMINARY EXAMINATION.

1084. Boil a portion of the substance for several minutes with KHO-solution. Then add Na_2CO_3-solution as long as it causes any precipitate, and boil again for several minutes. Filter, make the cold filtrate just acid with HCl, filter if necessary, and test separate portions as follows :—

Reagent added.	Result.	Inference.
1. $FeSO_4$ solution, freshly prepared	Blue precipitate . .	Presence of ferro- or ferricyanide.
	White precipitate .	Probable presence of cobalticyanide.
2. Fe_2Cl_6 solution . . .	Blue precipitate .	Presence of $(FeCy_6)^{iv}$
	Blood-red coloration	Presence of $(CyS)'$.
3. Add $ZnSO_4$ solution as long as it causes any precipitate	Light brown precipitate .	Presence of $(FeCy_6)'''$.
	White precipitate .	Presence of $(FeCy_6)^{iv}$, $(CoCy_6)'''$.
The precipitate produced by $ZnSO_4$ should be filtered off and fused into a colourless borax bead.	A blue bead is produced . .	Presence of $(CoCy_6)'''$.

1085. If Cyanide only is Present, the ordinary course of analysis is pursued, but it will be necessary to remove HCy by boiling the solution for some time after adding an acid (1001, 1007).

1086. **If Sulphocyanide only is Found** by the preliminary tests (1084), it may usually be decomposed in the portion of the substance which is to be examined for metals, by pouring upon it some strong HNO_3 in a porcelain dish and boiling down nearly to dryness. The liquid is then diluted and boiled, and may be considered as Solution III. (1001), any undissolved residue being treated as is there directed.

The less simple methods in paragraph 1088 may also be employed.

1087. **If the Presence of Ferro-, Ferri-, Cobalti-, Chromi-, or Mangano-cyanide** has been proved by the preliminary tests (1084), two methods of procedure are open to the analyst.

Either the cyanogen may be removed from the substance before commencing the analysis (1088); or the substance may be examined without any such preliminary treatment (1089).

The advantage of employing the more complicated method (1089) is that it enables the analyst to decide whether the metals are present in cyanogen acid-radicles or not. It also yields a more precise knowledge of the constitution of the substance.

1088. METHOD 1.—**The Removal of the Cyanogen from the Substance** may be effected by either of the following processes. The substance may then be examined for metals in the usual way.

1. Pour upon the powdered substance strong H_2SO_4 in a porcelain crucible, evaporate to dryness and ignite the residue strongly. When the residue is cold, dissolve it by heating it with a little strong HCl, then adding water and heating again.

2. Fuse the substance in a porcelain crucible with three or four times its weight of a mixture of three parts of Am_2SO_4 and one part of $AmNO_3$.

METHOD II

These lines run across both pages

1089. Boil the substance with water, filter, and wash the residu

<table>
<tr>
<td rowspan="4"><i>Filtrate</i>: examine this for metals, more especially the alkalis, and for acid-radicles according to the directions given for analysing a liquid (986)
The acid-radicles should be first tested for in a portion of the solution, and if $(FeCy_6)^{iv}$, $(FeCy_6)'''$, $(CoCy_6)'''$, one or more be found, the solution must be evaporated to dryness with HNO_3, and the residue strongly heated (General Table 1007) after filtering from the H_2S precipitate, in order to destroy these cyanogen radicles The Fe, Co, &c, in the cyanogen acid-radicles will then be detected in the General Table (1007).</td>
<td colspan="3"><i>Residue</i>: boil with KHO solution for several minutes, then add and boil again, filter and wash the residue :—</td>
</tr>
<tr>
<td colspan="3"><i>Filtrate</i>. pass H_2S, and if it causes any precipitate continue KHO*, heat and filter :—</td>
</tr>
<tr>
<td colspan="2"><i>Precipitate:</i> wash well with boiling water, and boil the precipitate with strong HNO_3, a black residue of HgS may remain, filter this off after diluting the acid, and confirm the presence of Hg in the precipitate by heating it with Na_2CO_3 in a bulb-tube (282)
The filtrate (or solution if HgS is absent) is evaporated to dryness, the residue is then dissolved in a little hot strong HCl, the solution is much diluted, saturated with H_2S, and filtered :—</td>
<td rowspan="2"><i>Filtrate:</i> add dilute saturation and filter :—
<i>Precipitate</i>: boil with KHO, filter off any black precipitate and examine it for Hg by heating it with Na_2CO_3 in a bulb-tube (282).
Add to the filtrate or solution HCl until it is acid, pass H_2S to saturation, and examine for As, Sb, Sn, (Pt, Au), by Table II, commencing at 1026.</td>
</tr>
<tr>
<td><i>Precipitate</i> examine for Pb and Cu by Table II (1025).</td>
<td><i>Filtrate</i> add AmCl, AmHO in excess, and Am_2S, and examine for Zn, Mn, Ni, Co, &c (1064)</td>
</tr>
</table>

* If H_2S causes a precipitate, NaHS or KHS may be added, drop by filtrate; this may be done instead of passing H_2S to saturation and then

(Fresenius).

with boiling water, adding the washings to the filtrate.

<table>
<tr><td colspan="4">some Na_2CO_3 solution as long as it causes any precipitate in the solution or the filtrate,</td></tr>
<tr><td colspan="3">to pass the gas until the liquid is saturated, then add more</td><td rowspan="5"><i>Residue</i> dissolve and examine for metals in the usual manner (1007)
Fe, Co, Mn, Cr, if found in this residue, were not present in cyanogen acid-radicles</td></tr>
<tr><td colspan="3">HNO_3 gradually until the liquid is just acid, pass H_2S to</td></tr>
<tr><td colspan="3"><i>Filtrate:</i> Divide into two parts, <i>α</i>, <i>β</i> —</td></tr>
<tr><td rowspan="2">α. Examine for acid radicles in the usual way, testing for $(CoCy_6)'''$ by adding excess of $ZnSO_4$, filtering and trying whether the precipitate gives a blue bead with borax.</td><td colspan="2">β. Evaporate to dryness and fuse the residue: when cold boil it with water and filter.—</td></tr>
<tr><td><i>Residue:</i> dissolve in HCl, and test for Al, Fe, Mn, Co (1064), the last three if found were present as cyanogen acid-radicles.</td><td><i>Filtrate</i> acidify a portion if yellow, with H$\bar{A}$ and add Pb$\bar{A}_2$, a yellow precipitate shows $(CrO_4)''$, the Cr having been present as cyanogen acid-radicle.
Test another part for Al by adding HCl in excess, then AmHO in excess.</td></tr>
</table>

drop, until it no longer gives any precipitate in the liquid or in the adding KHO.

1090. GENERAL GROUP TABLE FOR

Group I.—Reagent HCl.	*Group II.*—Reagent H_2S.
The Group Pp may contain in addition to $AgCl$, Hg_2Cl_2, $PbCl_2$:— *TlCl* (*white*). H_2WO_4 (,,) *TlCl* will usually be readily detected by a spectroscopic examination of the group pp. It may be removed from the group pp. by boiling with a little water, and pp^d from the cold filtrate by KI (Pb is also thus pp^d.): the pp is yellow, and gives the thallium spectrum. H_2WO_4: a white pp. becoming yellow on boiling. Its presence is confirmed by dropping a piece of Zn into a portion of the acid liquid and pp., when a *deep blue* colour will be obtained. Also by fusion of the pp. in a microcosmic bead, which will be *colourless* or *yellow* in the outer flame, *blue* (or with $FeSO_4$, *blood-red*) in the inner flame.	*The Group Pp may contain as sulphides, in addition to Hg, Pb, Bi, Cu, Cd:—* *Pd* (*black-brown*) *Tl* (*black*) [*Os*, *Rh*, *Ru* *] Insoluble in Am_2S. *And in addition to SnS, SnS_2, Sb_2S_3, As_2S_3, Au_2S_3, PtS_2:—* [*Ir*-sulphide*] *Mo*- ,, (*brown* *Se*- ,, (*red-yellow*) *Te*- ,, (*black*) Soluble in Am_2S. The *yellow* colour of a solution containing V is changed to *blue* by H_2S. *Tl* is readily found by the spectroscope in the group pp. *Pd* will remain in solution in excess of AmHO with Cd and Cu in Table II.A: from this solution it is pp^d. by adding HCl: its presence may be further confirmed by the $HgCy_2$ or KI tests (350, 351). The examination of the sulphides in Group II.B when the above elements may be present is best commenced by fusion with Na_2CO_3 and $NaNO_3$. From the fused mass, water dissolves sodium-arsenate, -molybdate, -selenate, and -tellurate, leaving SnO_2, sodium-antimonate, Au, Pt, and Ir undissolved; the elements may then be detected by special tests. For the separation and detection of the platinum metals a special work must be consulted. * Only completely pp^d if the liquid has been warmed, and H_2S passed for a long time.

PRECIPITATION OF RARER ELEMENTS.

Group III —Reagents AmCl, AmHO, and Am$_2$S.	Precipitated from the filtrate from Group III. by excess of HCl.	*Group V.*
The Group Pp may contain as sulphides, in addition to Fe, Zn, Mn, Ni, Co.— *U* (*black brown.*) *In* (,, ,,) *Tl* (,, ,,) *And as hydrates in addition to Al, Cr (and phosphates, &c.)*— *Ti* (*white, flocculent.*) *Be* (*colourless, gelatinous*). [*Zr* ,, ,,] [*Ta, Nb, Ce, La, Di, Y, E, Th*] *Tl* and *In* will usually be readily detected by a spectroscopic examination of the pp. *Tl* may be first separated and detected by dissolving some of the group pp. in boiling dilute HCl, and reducing any Fe present in the solution by H_2SO_3; then precipitating Tl by addition of KI, and testing the pp. by the spectroscope. A more complete scheme for the analysis of this group pp. will be found in par. 1091.	*As sulphides*:— *V* (*black*). *W* (trace, *brown*). *Ni* (trace, *black*). This pp. is dried and then fused with a mixture of Na_2CO_3 and KNO_3: on boiling the mass with water, NiO remains undissolved; it is filtered off, and its presence confirmed by fusion in a borax bead. The aqueous solution may contain alkaline vanadate and tungstate. V is separated by saturating the liquid with AmCl, and its presence confirmed in the pp. by the borax bead, and by dissolving the pp. in HCl and employing the reactions with H_2O_2 and with Zn (275, 273) The filtrate, after concentration if necessary, is acidified with HCl, and the presence of W confirmed by the Zn reaction and the microcosmic bead (397, 400).	The filtrate, after the separation of all the group precipitates, may contain besides Mg, K, and Na:— *Li, Cs, Rb.* These metals are readily detected by their very characteristic spectra (116). LiCl may also be detected by its behaviour with Na_2HPO_4. Before proceeding to the spectroscopic examination, it is well to convert the metals into chlorides if they are not already in that form. If the chlorides are evaporated to dryness and extracted with absolute ether and alcohol, to which a few drops of HCl have been added, LiCl, RbCl, CsCl dissolve, whilst the greater part of the KCl and NaCl remains undissolved. The solution is evaporated to dryness, dissolved in a little HCl, and Rb and Cs are pp^{d}. by $PtCl_4$ · this pp. is boiled with small quantities of water until it no longer gives the K spectrum, the spectra of Rb and Cs will then be seen, if present. Li is found in the filtrate from the $PtCl_4$ precipitate.

These lines run across both pages.

1091.—TABLE FOR ANALYSIS OF GROUP III. PRECIPITATE,

The precipitate may contain Fe, U, In, Tl, Al, Cr, Zn,

<table>
<tr><td colspan="5">After a preliminary spectroscopic examination has been made of a small portion of the the General Table (1090), the group pp. is dried and fused in platinum with $KHSO_4$ for long time with cold water:—</td></tr>
<tr><td rowspan="6">Residue. may contain Ta_2O_5, Nb_2O_5, also SiO_2, and traces of Fe and Cr which have escaped solution By fusion with $KClO_3$ and NaHO, Cr and Si are rendered soluble in dilute NaHO; from the residue, insoluble in NaHO, Ta_2O_5 and Nb_2O_5 may be removed by washing with dilute Na_2CO_3 solution.</td><td colspan="4">Solution reduce Fe by adding H_2SO_3 in excess, dilute consider- covering the vessel, and by passing a stream of CO_2</td></tr>
<tr><td rowspan="5">Pp. may consist of TiO_2, possibly also traces of Zr.
Confirm the presence of TiO_2 by the microcosmic bead (267).</td><td colspan="3">Filtrate: add a few drops of strong HNO_3, and precipitate once more by adding excess</td></tr>
<tr><td colspan="3">Pp. may contain Fe, In, U, Cr, Al, Be, &c.</td></tr>
<tr><td colspan="3">Pp. may contain Fe, In, U, &c Dissolve large excess of HCl, add $BaCO_3$, and let</td></tr>
<tr><td rowspan="2">Pp. may contain Fe, In, U, also traces of Al and Cr.
It is dissolved in HCl, and $NaHCO_3$ is added in excess, whereby U alone is obtained in solution.
In is found by the spectroscope:
Or by fusion with Na_2CO_3 and $KClO_3$.</td><td colspan="2">Filtrate may contain Th. Ba is $pp^{d.}$ by H_2SO_4, the liquid exactly neutralised of K_2SO_4 are added, allowed to stand for pp. is washed with</td></tr>
<tr><td>Pp may contain Zr, Th, Ce.
Th and Ce are dissolved away by boiling with dilute HCl, and are $repp^{d.}$ by AmHO.</td><td></td></tr>
</table>

WHEN THE RARER ELEMENTS MAY BE PRESENT.

Mn, Ni, Co, Ti, Be, [Zr, Ta, Nb, Ce, La, Di, Y, E, Th].

<table>
<tr><td colspan="3">pp. for Tl and In, and Tl has been further tested for, if necessary, by KI as directed in
some time, the cold mass is then powdered and allowed to stand, with shaking, for a</td></tr>
<tr><td colspan="3">ably and boil for a long time, preserving the liquid from oxidation by the air by
unless it smells constantly of SO_2:—</td></tr>
<tr><td colspan="3">concentrate by evaporation, and add AmHO in excess: filter, dissolve the pp. in HCl,
of AmHO :—</td></tr>
<tr><td colspan="2">Dissolve it in HCl, and add excess of cold strong KHO solution :—</td><td rowspan="4"><i>Filtrate</i> may contain.—
Zn, Mn, Ni, Co.</td></tr>
<tr><td>in HCl, boil off any stand :—</td><td rowspan="3"><i>Filtrate</i> may contain Al, Cr, Be: dilute and boil for some time, filter. Al remains in solution, and may be pp$^{d.}$ by adding HCl just in excess, then AmHO in excess.
Cr and Be are separated by fusion with Na_2CO_3 and $KClO_3$, dissolving in water and pp^{s}, Be by adding HNO_3 in excess, then AmHO in excess.</td></tr>
<tr><td>Zr, Ce, La, Di, V, E, adding excess of is concentrated and with KHO. Crystals and the liquid is some hours The K_2SO_4 solution.</td></tr>
<tr><td><i>Sol^{n}.</i> may contain Y, E, also Be.
The solution is pp$^{d.}$ by AmHO, and Be dissolved away from it by $H_2C_2O_4$.</td></tr>
</table>

GENERAL METHODS FOR DETECTING ORGANIC SUBSTANCES.

The constituent elements of an organic substance may be detected by the processes mentioned in paragraphs 631–643.

In the following scheme provision is made for detecting the organic substances whose reactions are given in paragraphs 647–892.

INTRODUCTORY REMARKS.

1092. **The Detection and Identification of an Organic Substance** is usually more difficult than the detection of an inorganic substance. This is especially the case when a mixture of organic substances has to be dealt with.

Since no complete scheme of separation is possible, recourse must be had to several partial separations, such as the precipitation of one or more of the substances in an insoluble form, or the use of a suitable liquid which is a solvent for certain substances but not for others. Sometimes the substances may be separated by the application of heat, the more volatile substance passing off as vapour and leaving the non-volatile body behind: or by fractional distillation (646a).

The suggestions and Tables which are given below include:—(1) The preliminary tests such as the action of heat, acids and other reagents; (2) the identification of the elements present; (3) the identification of the compounds by their special properties.

It should be remembered that the student must be familiar with the reactions and properties of organic bodies given in pars. 647–892 before he can apply these as tests.

SCHEME FOR THE DETECTION OF ORGANIC SUBSTANCES.

Preliminary Examination.

1093. **Notice the Physical Properties of the Substance,** such as its colour and smell; also whether it is solid or liquid, and, if solid, whether it is amorphous or crystalline. The determination of the solubility in various reagents (1098), and of the melting-point (645), boiling-point (646), and specific gravity (644), is often of great assistance.

Note also whether the substance or its solution is acid to litmus. Acid reaction usually indicates the presence of salts of certain bases, *e.g.*, urea nitrate · esters also usually contain free acid.

1094. **Ascertain what Changes the Substance undergoes when it is Heated,** whether it is volatile or fusible, and whether any gases or vapours are evolved.

The test is made by heating a small quantity of the substance in a test-tube, so as to prevent any considerable change from oxidation occurring by free contact with air. The results which may be obtained are tabulated on the following page.

It is well to repeat this test by heating the substance to a high temperature in free contact with the air (1095). The result thus obtained indicates whether any non-volatile and incombustible substance is present, since this will remain as a residue. This residue will usually consist of metal, either free or as oxide or carbonate; and the examination for the metal is easily effected by applying the ordinary analytical methods to this residue.

EFFECT OF HEATING THE SUBSTANCE IN A TEST-TUBE.

Result.	Observation.	Inference.
a. The substance volatilises leaving no residue.	The vapour is non-inflammable.	Chloroform.
	The vapour burns with a pale blue flame	Alcohol, aldehyde
	The vapour burns with a blue flame.	Chloral, carbon disulphide
	The vapour burns with a pale blue flame; slight charring occurs	Glycerol.
	The vapour burns with a smoky flame.	Aniline, benzene, turpentine
	The vapour burns with a luminous flame.	Ether, ethyl acetate, benzaldehyde, or nitrobenzene
	The vapour is acid and pungent and inflammable	Formic acid, or acetic acid.
b The substance fuses, and volatilises without residue	The vapour burns with a smoky flame	Phenol
	NH_3 is smelt	Urea.
	Pungent fumes yielding a white sublimate.	Oxalic acid, or succinic acid.
	Pungent fumes, burning with a smoky flame; and sublimate formed	Benzoic acid.
	A slight explosion.	Picric acid
c. The substance fuses and chars.	It chars slowly, evolving CO, &c	Formate.
	It chars slowly, evolving acetone	Acetate.
	It emits a smell of charred sugar.	Tartaric acid, citric acid, malic acid, pyrogallol, all sugars, meconic acid.
	It emits a smell of burnt wool, and NH_3	Urate, albumen, or gelatin.
	It evolves phenol, which burns with a smoky flame.	Salicylate.
	Violet vapour of I.	Iodoform.
	White crystalline sublimate.	Hydroquinone
d. The substance chars without fusing.	A carbonate remains.	Succinate
	It emits a smell of burnt sugar.	Starch, cellulose.
	It evolves orange-coloured vapour.	Tannate.
	It evolves red vapour.	Gallate.
e The substance neither chars nor fuses.	A carbonate remains (505).	Oxalate.
f. The substance fuses to a coloured liquid.	The liquid is reddish; vapour of unpleasant smell is emitted, which burns with a smoky flame.	Hippurate, or an alkaloid.
g The substance fuses, and evolves cyanogen.	No further special observations.	Cyanide.
	A smell of H_2S is also noticed.	Sulphocyanide
	The substance darkens in colour.	Ferrocyanide, or ferricyanide.

1095. Ascertain whether any Metals are Present by heating the substance on a piece of thin porcelain; if a black residue of carbon is left, burn off the carbon by heating it strongly in the blowpipe-flame (Exp. 81, **105**). One of the following results may be obtained.

(*a*) *A Coloured Residue* may remain and will probably be due to the presence of a metal which produces a coloured oxide. The residue may be examined directly by paragraph **991**, and then after solution (**1001**) by the General Table (**1007**).

(*b*) *A White Residue* may remain, which when it is moistened with water shows an alkaline reaction to test-paper: this may be caused by the presence of K, Na, Ba, Sr, Ca, or Mg in an organic salt. The residue may be examined by the preliminary tests, **990**, **991**, and may then be dissolved (**1001**) and examined by the General Table (**1007**).

1096. Mix the Substance with Powdered Soda-lime and heat. Note particularly any of the following changes.

(*a*) *A Smell of Ammonia*, which is evolved from nitrogenous substances, such as ammonium salts, urea, albuminous bodies, alkaloids, urate and hippurate.

Nitrocompounds, such as nitrobenzene and picrate, do not give off ammonia when they are heated with soda-lime.

(*b*) *Aniline*, evolved from aniline compounds.

(*c*) *Benzene*, evolved from benzoate or hippurate.

(*d*) *Phenol*, evolved from salicylate.

1097. Try the Action of Strong Sulphuric Acid upon the Substance, both in the cold and on heating: the results which may be obtained are tabulated on page 422.

Note.—This test may be supplemented by heating the substance with *dilute* H_2SO_4, since formates, acetates and cyanogen-compounds may be frequently detected by this means.

Effect of adding Strong H_2SO_4 to the Substance, or to its Strong Solution, and then Heating.

Result.	Observation.	Inference
a. No change occurs in the cold, and either little or no charring on heating.	CO evolved; no charring.	Formate.
	$H\bar{A}$ evolved, no charring	Acetate
	CO and CO_2 evolved, no charring	Oxalate.
	Pungent fumes; no charring.	Chloral, chloroform
	Slowly darkens; irritating fumes evolved	Succinate, or benzoate.
	Slowly darkens; acid fumes, then CO and CO_2 evolved	Malate.
	Slowly darkens, CO and CO_2 evolved. (Difference from tartrate, &c)	Citrate.
	Slowly darkens; pungent fumes evolved	Urea.
	Effervesces; slowly darkens.	Urate
	HCN evolved, no charring	Cyanide.
	HCN and finally CO evolved, white precipitate	Ferricyanide.
	CO evolved, and liquid becomes turbid	Ferrocyanide.
	SO_2 evolved, yellow S formed	Sulphocyanide
	Slight charring.	Meconic acid
b No change occurs in the cold, but the substance turns dark-coloured, or chars, when it is heated	Fragrant smell; vapour burns with a smoky flame, SO_2 is evolved, and charring occurs (Confirm by 734)	Alcohol
	Acrolein is smelt, and ultimately SO_2 is evolved with much charring	Glycerol.
	CO and SO_2 are evolved, with gradual charring.	Glucose.
	CO, CO_2, and SO_2 are evolved, with rapid charring.	Tartrate
	SO_2 is evolved, and charring occurs	Hippurate.
	The substance then turns reddish-brown, evolving CO_2 and pungent fumes	Salicylate
	The substance turns brown, and SO_2 is evolved	Tannate.
	A vivid red coloration is produced, and SO_2 is evolved	Gallate.
	Characteristic smell, then charring, and evolution of SO_2	Turpentine.
c. The substance becomes coloured in the cold; the colour deepens on heating.	Pale brown, cold, blackening on heating	Quinine, cinchonine, morphine, or strychnine.
	Yellow, cold, puce, on heating	Narcotine.
	Pink, cold; blackening on heating	Brucine.
	Yellow, cold, charring on heating, and evolving CO and SO_2	Starch.
	Brown, cold; charring rapidly, and evolving CO and SO_2 Solid cane-sugar gives these changes slowly.	Cane-sugar.
	Chars at once, forming white aniline sulphate	Aniline.

1098. Test if the Substance is Soluble in or Miscible with :—(1) Cold water, (2) hot water, (3) alcohol, and (4) ether.

Valuable information can frequently be obtained from this treatment with solvents, since it affords a ready means of effecting the separation of substances from one another if a suitable solvent can be found. The solubility of many organic substances is stated in the following Table:—

SOLUBILITY OF ORGANIC SUBSTANCES IN WATER, IN ALCOHOL AND IN ETHER.

s = soluble, *i* = insoluble, *ss* = slightly soluble, *vs* = very soluble, and *vss* = very slightly soluble.

NAME OF SUBSTANCE.	WATER		ALCOHOL.	ETHER.
	COLD.	HOT.		
ACIDS—				
Formic,	vs	vs	vs	i
Acetic,	vs	vs	vs	s
Oxalic,	s	vs	vs	s
Succinic,	ss	vs	s	ss
Benzoic,	ss	s	s	s
Malic,	vs	vs	vs	vss
Tartaric,	s	s	s	i
Citric,	s	s	s	i
Salicylic,	ss	s	s	vs
Tannic,	s	s	ss	vss
Gallic,	ss	s	s	s
Meconic,	ss	s	s	vss
Uric,	vss	vss	ss	i
Hippuric,		s	s	ss
Picric,	vss	s	s	s
ALKALOIDS—				
Quinine,	i	i	vss	ss
Cinchonine,	i	i	ss	vss
Morphine,	vss	vss	s	vss
Strychnine,	i	i	s (hot)	i
Narcotine,	i	i	ss	s
Brucine,	ss	ss	s	i
Caffeine,	ss	s	ss	vss
HYDROCARBONS—				
Turpentine,	i	i	vs	vs
Benzene,	i	i	s	s
HALOID COMPOUNDS—				
Chloroform,	i	i	s	s
Iodoform,	i	i	s	s

NAME OF SUBSTANCE.	WATER.		ALCOHOL.	ETHER.
	COLD.	HOT.		
ALCOHOLS AND PHENOLS—				
Methyl alcohol,	vs	vs	vs	vs
Ethyl alcohol,	vs	vs	vs	vs
Glycerol,	s	s	s	i
Phenol,	s	s	vs (hot)	∞
Hydroquinone,	s	s	s	s
Pyrogallol,	vs	vs	vs	s
ALDEHYDES—				
Formaldehyde,	ss	ss	s	i
Acetaldehyde,	vs	vs	s	s
Chloral,	s	s		..
Benzaldehyde,	s	s	vs	vs
AMIDO-COMPOUNDS—				
Urea,	vs	vs	vs	ss
Aniline,	vss	vss	s	s
MISCELLANEOUS—	s	s	s	s
Ether,	ss	ss	vs	vs
Ethyl acetate,	vs	vs	vs	vs
Acetone,	s	s	s	s
Carbon disulphide,	i	i	vs	vs
Nitrobenzene,	i	i	s	s
CARBOHYDRATES AND ALBUMINOUS COMPOUNDS—				
Dextrose,	vs	vs	s	s ($CH_3.OH$)
Levulose,	s	s	s	
Cane-sugar,	vs	vs	ss	i
Maltose,	s	s	s	i
Milk sugar,	ss	ss	vss	i
Cellulose,	i	i	i	i
Starch,	i	s	i	i
Dextrin,	s	s	i	i
Albumen,	s	s	i	i
Gelatin,	i	s	i	i

s=soluble i=insoluble, ss=slightly soluble, vs=very soluble, vss=very slightly soluble.

SPECIAL EXAMINATION OF ORGANIC SUBSTANCES.

1099. If the substance is a simple one, it will probably have been identified and it will only be necessary to apply confirmatory tests. But if the preceding treatment has given any indication that the substance is a mixture, some method of separation should be devised. For a solid the action of solvents (1098) may be utilised, or treatment with solutions of acids or of alkalis may be successful. A liquid may be subjected to fractional distillation (646a). In any case it is advisable to ascertain what elements are present in the substance.

The following experiments will indicate the presence of the more important elements.

1100. Ascertain what Elements are present by heating the substance with a little sodium in an ignition-tube until it is charred. Then drop the tube into a few c.c. of water contained in a mortar. Grind up the fragments with the water, filter, and divide the filtrate into three portions.

(*a*) To the first portion add a small crystal of $FeSO_4$, boil the liquid and acidify it with HCl; a blue or green coloration shows the presence of *nitrogen* (637).

(*b*) To the second portion add a small crystal of sodium nitroprusside; a violet or purple colour shows the presence of *sulphur* (638).

(*c*) If the substance contains no nitrogen, to the third portion add HNO_3 and $AgNO_3$-solution; a white or yellow precipitate shows the presence of a *halogen* (643).

(*d*) If the substance contains nitrogen, test for the halogen by igniting a fresh part of the substance with excess of pure lime in a combustion tube. Shake out the contents of the tube into dilute nitric acid, filter the liquid and add $AgNO_3$-solution; a white or yellow precipitate shows the presence of a *halogen* (643).

The further examination of the substance will depend upon what elements have been detected by the above tests.

1101. Hydrolysis may be applied to certain substances such as esters, alkyl cyanides, glucosides and sugars, which may by this means be split up into readily recognised products. The process consists in the prolonged heating with a moderately strong acid, such as HCl or H_2SO_4, or with KOH-solution. The process may be carried out in a flask connected with a reflux condenser.

The resultant products include alcohols, glucoses and acids, and these may be detected by the methods given under the separate tests and reactions.

1102. The Presence of an Alkaloid may be ascertained by the application of the general tests given in paragraphs 735–738. The identification of the particular alkaloid present may usually be effected by carefully extracting with warm pure alcohol acidified with dilute HCl, driving off the excess of alcohol, diluting the extract with water and testing the solution by paragraphs 739–785.

1103. The Presence of Organic Acids or their Salts can usually be determined by the formation of characteristic insoluble compounds with the metals iron, calcium, or silver (1104, 1105, 1106).

The acid-radicles should be present in sodium, potassium, or ammonium salts, since other metals interfere more or less seriously with their detection by these tests.

(*a*) If alkali-metals only are present, the solution to be tested must be rendered neutral or *very faintly* alkaline. The most suitable reagent to be added for producing this condition is dilute HNO_3, or sodium hydrate solution. According to the reaction of the solution, one or other of these reagents is used, and is added until the solution does not affect the colour of test-papers.

(*b*) If metals other than the alkali-metals are present, the substance must be boiled with excess of Na_2CO_3-solution, filtered, and acidified with HNO_3; the CO_2 is then boiled off and the solution is carefully neutralised with NaHO.

(*c*) If metals of Group II. or III. are present, the following treatment may be requisite in order to ensure their removal. The solution is acidified, if it is not already acid, with dilute HCl, and H_2S is passed to saturation; the liquid is then filtered, and AmHO and Am_2S are added to the filtrate: the liquid is then filtered again and the filtrate is reserved. The filtrate will contain the organic acid-radicles in ammonium salts, together with excess of Am_2S. Its further treatment will depend upon whether other metals (Ba, Sr, Ca, Mg) are present.

If no other metals are present, acidify the liquid with dilute HCl, boil off the liberated H_2S, filter if necessary, and neutralise the filtrate with NaHO. This liquid may now be tested with the reagents mentioned in pars. **1104**, **1105** (see *Note* below).

If other metals are present, acidify with dilute HCl, boil off the H_2S, add Na_2CO_3-solution in excess, filter, acidify with HNO_3, boil off CO_2, and carefully neutralise with NaHO: then proceed to test the liquid by pars. **1104**, **1105**.

Note.—A separate solution of the original substance must be prepared for the $AgNO_3$ test (1106), in which dilute HNO_3 is used instead of HCl, else the $AgNO_3$-solution will give a white precipitate of AgCl which may be mistaken for an organic salt.

The results given by the tests in pars. **1104**–**1106** with very dilute solutions are not pronounced, and are in some cases not even visible, and strong solutions should always be employed.

It will be seen that the results which are yielded by other substances besides the organic acid-radicles are given in paragraph **1104**.

1104. ACTION OF $FeCl_3$ SOLUTION ON NEUTRAL SOLUTION OF ORGANIC SUBSTANCES.

Result	Observation.	Inference.
a. A red coloration is produced in the cold	The coloration is destroyed by $HgCl_2$ solution, and is unaltered by dilute HCl.	Sulphocyanide.
	The coloration is destroyed by dilute HCl, a red precipitate forms on boiling	Formate, or acetate.
	(Diff.) In the original solution $AgNO_3$ gives a black precipitate in hot solution	Formate.
	(Diff.). Strong H_2SO_4 gives a smell of $H\bar{A}$	Acetate.
	The coloration is with difficulty destroyed by HCl $CaCl_2$ produces a white precipitate in the original solution on boiling (Diff. from formate and acetate)	Meconate.
	Coloration destroyed by $H\bar{A}$, but not by HCl.	Pyrogallol.
b. A purple coloration is produced in the cold.	Purple colour not destroyed by $H\bar{A}$. A salicylat when heated with strong H_2SO_4 and CH_3OH gives off methyl salicylate (Diff. from phenol)	Salicylate.
	Purple colour destroyed by $H\bar{A}$. Phenol solution when treated with AmHO and bleaching-powder turns blue (Diff from a salicylate	Phenol.
c. A blue-black precipitate is produced in the cold.	The precipitate disappears on boiling With a gallate KCN produces a red coloration. (Diff. from a tannate.)	Gallate.
	The precipitate is permanent on heating. Ammoniacal $CuSO_4$ solution produces a green precipitate with a tannate. (Diff from a gallate.)	Tannate.
d. A blue precipitate is produced in the cold.	The colour of the precipitate is changed by KHO into brown.	Ferrocyanide.

Result	Observation	Inference.
e. A buff or reddish precipitate is produced in the cold	The addition of HCl gives crystalline scales of benzoic acid. The original substance burns with a smoky flame. (Diff from a succinate.)	Benzoate.
	The precipitate is soluble in HCl. A succinate is precipitated on the addition of $BaCl_2$, AmHO, and alcohol. (Diff from benzoate.)	Succinate.
	On the addition of HCl, hippuric acid separates. The original substance burns with a smoky flame, and evolves NH_3 and C_6H_6 when it is heated with soda-lime	Hippurate.
	The reddish precipitate dissolves in HCl. Confirm by adding to the original substance $K_2Cr_2O_7$ and H_2SO_4, a blue colour appears.	Aniline.
f. A coloration is produced in the cold.	Brown or olive-green coloration. Confirm by adding $FeSO_4$ solution to the original substance, a blue precipitate forms.	Ferricyanide.
	The coloration is blue.	Morphine.
g. No effect is produced until the mixture is heated.	Reddens on heating	Quinine, caffeine, brucine.
	Red precipitate on heating.	Cinchonine, or strychnine.
	Slight red precipitate on heating.	Narcotine.

1105. ACTION OF $CaCl_2$ SOLUTION ON NEUTRAL SOLUTION OF ORGANIC SALTS.

Result	Observation.	Inference.
a. A white precipitate forms in the cold.	The precipitate is insoluble in $H\bar{A}$ and in KHO; when dried and heated, it scarcely chars and leaves a carbonate	Oxalate.
	The precipitate is soluble in HA and in KHO, it often only appears on shaking, and chars when dried and heated Confirm by heating the original solution with ammoniacal $AgNO_3$ (694, Note)	Tartrate.
	Add $Pb\bar{A}_2$ to the original solution, a white precipitate forms	Meconate.
	The precipitate is decomposed by HCl, with separation of uric acid	Urate.
	The precipitate is unchanged when heated	Hippurate, benzoate, or tannate.
b. A white precipitate forms on heating.	The precipitate is soluble in $H\bar{A}$ and insoluble in cold KHO, it chars on being dried and heated	Citrate.
	The precipitate forms more readily in the presence of alcohol, it chars on being heated Confirm by adding $Pb\bar{A}_2$ to the neutral solution, and fusing the precipitate under water	Malate
c. A white precipitate forms in the presence of alcohol.	Confirm by treating the neutral solution with $FeCl_3$, a reddish precipitate forms.	Succinate.

1106. ACTION OF $AgNO_3$ SOLUTION ON NEUTRAL SOLUTION OF ORGANIC SALTS.

Result	Observation	Inference.
a. A white precipitate forms in the cold.	The precipitate is unaltered when heated.	**Oxalate, meconate, salicylate, malate, or hippurate.**
	The precipitate dissolves when heated it is not formed in dilute solution	**Acetate.**
	The precipitate forms in strong solutions, rapidly turning black, in dilute solutions a black precipitate of Ag is formed when the liquid is heated.	**Formate**
	The precipitate turns grey when it is heated	**Citrate, or malate.**
	The precipitate slightly darkens when it is heated	**Succinate, or benzoate.**
	The precipitate rapidly turns black	**Tannate, gallate, urate, phenol, and hydroquinone.**
	The precipitate turns black when the liquid is boiled.	**Tartrate.**
	The precipitate is soluble in AmHO, and insoluble in dilute HNO_3	**Cyanide or sulphocyanide**
	The precipitate is insoluble in AmHO, and insoluble in dilute HNO_3	**Ferrocyanide.**
	Immediate black precipitate.	**Pyrogallol.**
b. An orange precipitate is formed.	The precipitate is soluble in AmHO, and insoluble in dilute HNO_3	**Ferricyanide**

EXAMPLE SHOWING HOW TO ENTER THE RESULTS OF ANALYSIS OF A COMPLEX SUBSTANCE.

1107. The substance given for analysis consisted of a powder containing pink, blue, white, and black particles. It smelt faintly of ammonia.

PRELIMINARY EXAMINATION FOR METALS.

Experiment.	Observation.	Inference.
EXPT. I.—Heated in a small dry test-tube.	Water given off which turned turmeric-paper brown.	Pres. of H_2O. Pres of NH_4.
	Strong smell of NH_3 gas	Pres. of NH_4.
	Substance blackened, no smell of burning.	Prob. pres of Co, Cu, and abs of $\bar{T}$ and $\bar{A}$.
	Slight white sublimate.	Pres. of As, NH_4, Hg.
Confy.—Held in the upper part of the tube a glass rod with a drop of lime water hanging on its end.	The lime water became milky.	CO_2 evolved.
	Brown nitrous fumes evolved, recognised by their smell.	Pres of nitrate.
	Cl-gas evolved, found by smell and bleaching litmus	Pres. of Cl.
Confy.—Boiled a portion of the substance with KHO solution.	NH_3 gas was evolved, recognised by its smell, and by giving white fumes with strong HCl.	Pres of NH_4.
Confy.—Heated strongly another portion of the dried substance with Na_2CO_3 in a bulb-tube.	No mirror formed.	Abs. of Hg and As.
EXP. II.—Heated a portion of the substance on a loop of platinum wire in the Bunsen flame, moistened with HCl and heated again in the flame	*Bright yellow* flame. The flame appeared *crimson* through the indigo-prism.	Pres. of Na. Pres. of K.
Heated for some time in the tip of the inner blowpipe flame, moistened with HCl, and again held in the Bunsen flame.	*Crimson* col^r. appearing *intense red* through the indigo-prism. *Bright green* col^r. with *blue* core.	Pres. of Sr. Pres. of Cu.

Expt	Observation	Inference.
Expt. III.—Heated a portion of the substance on charcoal in the inner blowpipe flame.	The greater part of the substance fused readily, and was absorbed by the charcoal	Pres. of a salt of K, Na.
	Deflagration occurred.	Pres. of chlorate, nitrate.
	A red metallic residue remained	Pres. of Cu.
Confy.—Fused in a clear borax bead in the outer and inner blowpipe flames	In the outer flame a bead *green* whilst hot, *blue* when cold In the inner flame *red and nearly opaque.*	Pres. of Cu.
Confy.—Fused on platinum foil with Na_2CO_3 and KNO_3.	No *bluish green* or *yellow* mass on cooling	Abs. of Mn and Cr
Confy.—Fused on charcoal in the inner blowpipe flame with Na_2CO_3.	Red metallic residue.	Pres. of Cu
	A portion placed on a silver coin and moistened gave no black stain	Abs. of S.

Preliminary Examination for Acid-Radicles.

Expt.	Observation.	Inference.
Expt. I.—Added dilute HCl without heating	A colourless gas was evolved, which was free from smell, and turned a drop of lime water milky.	Pres. of carbonate. Abs. of sulphite, hypochlorite, &c.
Heated to boiling.	Cl was evolved, recognised by its smell and by bleaching moist litmus-paper	Pres. of nitrate, chlorate, or some other oxidising substance
Expt. II.—Added strong H_2SO_4.	A bright yellow chlorous gas evolved, which crackled when warmed.	Pres. of chlorate.
Confy. — Dropped in copper turnings and heated.	Reddish brown fumes evolved.	Pres. of nitrate.
Heated strongly, cooled, and rinsed out	The tube when dry was seen not to be etched.	Abs. of fluoride.
Confy.—Boiled a portion of the substance with water, added strong H_2SO_4, cooled and poured in $FeSO_4$ solution carefully.	A brown ring formed on the surface of the acid.	Pres. of nitrate.

Examination for Metals in the Wet Way.

Boiled a portion of the substance with Fe_2Cl_6, $FeSO_4$ and KHO, added HCl in excess, no blue pp.;—*Absence of Cy.*

These lines run across both pages.

Boiled a portion of the substance with water; as it did not com-
upon the undissolved residue: effervescence occurred, and on boiling, Cl
Cl was perceived; the substance was completely dissolved. Cooled
previously to give no pp. on addition of a little dilute HNO_3:—

<table>
<tr><td rowspan="3"><i>No pp.</i>
<i>Abs of Group I</i>
Hg′, Ag, and prob. Pb</td><td colspan="2">Diluted with water and passed H_2S until the liquid smelt strongly</td></tr>
<tr><td rowspan="2"><i>A black pp</i>
Exam^d by Table II</td><td><i>Filtrate</i> which gave no further pp. with Boiled until it no longer smelt of H_2S, on addition of HNO_3 (<i>prob. pres of Fe</i>) of the HCl sol^n. to some $AmHMoO_4$ boiled; filtered —</td></tr>
<tr><td><i>A brown pp.</i>
Exam^d by Table III A</td></tr>
</table>

EXAM$^{N.}$ OF PP. IN GROUP II.—Removed from the filter into a porcelain dish and boiled with KHO, filtered:—

<table>
<tr><td rowspan="4"><i>Filtrate</i> — Acidified with HCl, a <i>white</i> milky liquid only. <i>Abs of Group</i> II B.</td><td colspan="4"><i>Pp</i> removed from <i>filter</i> by a glass rod into a porcelain dish and boiled with strong HNO_3 as long as any red fumes came off, added dilute H_2SO_4 and stirred well.</td></tr>
<tr><td rowspan="3"><i>No pp</i> — <i>Abs of Hg and Pb.</i></td><td colspan="3">Added to sol^n excess of AmHO blue solution (<i>Pres of Cu</i>) —</td></tr>
<tr><td rowspan="2"><i>No pp</i> — <i>Abs of Bi.</i></td><td colspan="2">Acidified the blue sol^n with HCl and saturated with H_2S, filtered off the black pp. rapidly and boiled it with dilute H_2SO_4.</td></tr>
<tr><td><i>Pp</i> dissolved in a little boiling dilute HNO_3, added AmHO in excess, then excess of $H\bar{A}$, then K_4FeCy_6, chocolate red pp.:— <i>Pres of Cu</i></td><td><i>Filtrate</i> diluted much and passed H_2S, no pp.:— <i>Abs. of Cd.</i></td></tr>
</table>

EXAM^N OF PP. IN GROUP III.A.

Dissolved in a little boiling dilute HCl, added pure NaHO in excess, boiled and filtered:—

<table>
<tr><td rowspan="2"><i>Filtrate</i> · added AmCl in excess, no pp:— <i>Abs. of Al.</i></td><td colspan="2"><i>Pp</i> dried and fused on platinum foil with Na_2CO_3 and KNO_3, boiled the colourless (<i>abs. of Cr</i>) mass when cold with water; decanted from the undissolved residue:—</td></tr>
<tr><td><i>Residue</i> in the dish dissolved by boiling with a little HCl, added several drops of KCyS: a blood-red coloration. <i>Pres. of Fe.</i></td><td><i>Sol^n</i> being colourless proved abs. of Cr.
Acidified with $H\bar{A}$, boiled, and added $Pb\bar{A}_2$, no yellow pp.:— <i>Abs. of Cr.</i></td></tr>
</table>

pletely dissolve, the sol^n was decanted, and a little dilute HCl poured
was smelt. Added a little strong HCl, boiled as long as any smell of
this sol^n and mixed it with the water sol^n which had been proved

<table>
<tr><td colspan="3">of the gas, filtered :—</td></tr>
<tr><td colspan="3">H_2S was pink (<i>prob. pres. of Co</i>)
added some strong HNO_3 and boiled to dryness· the colour of the sol^n changed to light brown
Warmed the residue with HCl, it dissolved completely (<i>Abs of</i> H_2SiO_3) Added a small portion
warmed, no pp.. <i>Absence of</i> $(PO_4)'''$. To the rem^r added AmCl then excess of AmHO, and</td></tr>
<tr><td colspan="3"><i>Filtrate</i> was again pink (<i>presence of Co</i>) : added much Am_2S and boiled, filtered —</td></tr>
<tr><td rowspan="2"><i>A black pp.</i>
Exam^d by Table III.B</td><td colspan="2"><i>Filtrate</i> was yellow (<i>absence of Ni</i>) added Am_2CO_3), filtered·—</td></tr>
<tr><td><i>A white pp.</i>
Exam^d by Table IV.</td><td><i>Filtrate.</i>
Exam^d by Table V.</td></tr>
</table>

EXAM^N OF PP. IN GROUP III.B.

Rinsed the pp. off the filter with some cold dilute HCl, stirred well, filtered .—

<table>
<tr><td rowspan="2"><i>Pp</i> was black and had already been proved to contain no Ni, fused a portion into a clear borax bead bead blue in both flames·—
<i>Pres of Co.</i></td><td colspan="2"><i>Filtrate</i> boiled until it no longer smelt of H_2S, added a crystal of $KClO_3$, boiled until the smell of Cl ceased, cooled and added pure NaHO in excess —</td></tr>
<tr><td>No pp
<i>Abs of Mn.</i></td><td>Passed H_2S into the sol$_n$
no pp —
<i>Abs of Zn.</i></td></tr>
</table>

EXAM^N OF PP. IN GROUP IV.

1. *Exam^n by Flame col^n*—Dissolved a small quantity of the pp. in a few drops of HCl upon a watch-glass, dipped a loop of Pt-wire into the sol^n and held it in the Bunsen flame : a crimson red colour was imparted to the flame, which appeared intense red through the indigo-prism : *Pres. of Sr.* This coloration was followed by a yellowish green very persistent col^n : *Pres. of Ba.* Confirmed by spectroscope.

2. *Exam^n in the Wet way.* Dissolved the rest of the pp. in as little boiling $H\bar{A}$ as possible; to a small part of the solution, perfectly cold, added $CaSO_4$-sol^n· an immediate pp. formed : *Pres. of Ba.*

To the remainder of the $H\bar{A}$-sol^{n}, proved to be acid to litmus, added K_2CrO_4 until the liquid appeared yellow, warmed and poured through a double filter :—

Pp., which contained all the Ba present, was rejected.	To the clear *filtrate*, which was orange red in colour, added AmHO until the colour changed to light yellow, then added Am_2CO_3 in excess and filtered :—	
	Filtrate was rejected.	*Pp.* · dissolved in as little boiling $H\bar{A}$ as possible; added to a small portion of the sol$^{n.}$ $CaSO_4$ sol^{n} and boiled, a pp. formed, showing *pres. of Sr.* To the remainder of the $H\bar{A}$ solution added H_2SO_4, boiled, filtered, and to the filtrate, which gave no pp. after being boiled with more H_2SO_4, added AmHO in excess and $Am_2C_2O_4$, no pp.. *Abs. of Ca.*

Exam$^{N.}$ of Filtrate from General Table for Group V.

Evaporated the filtrate to dryness in a porcelain dish, scraped out the residue upon platinum foil, and ignited strongly until white fumes ceased to appear : dissolved the residue off the foil by boiling with water to which several drops of HCl had been added, divided the sol$^{n.}$ into two unequal parts :—

To the larger portion added several drops of H_2SO_4 and boiled, then AmHO in excess and several drops of $Am_2C_2O_4$; on heating, a slight pp. formed ; filtered, and added to the clear filtrate Na_2HPO_4, a white crystalline pp. :— *Pres. of Mg.*	*Into the smaller portion* dipped a loop of platinum wire, and held it in the Bunsen flame, an intense yellow col^{r} :— *Pres. of Na,* appearing crimson through the indigo-prism :— *Pres. of K.* Confirmed the pres. of K by stirring a fresh portion of the sol$^{n.}$ with $PtCl_4$ on a watch glass, a yellow pp. formed.

Metals found :—Cu, Fe, Co, Ba, Sr, Mg, Na, K, NH_4.

EXAM$^{N.}$ FOR ACID-RADICLES.

The following acid-radicles have already been detected: $(CO_3)''$, $(NO_3)'$, $(ClO_3)'$.

By reference to the Table of Solubility (1038), under the columns corresponding to the above metals, the only salt insoluble in acids is found to be $BaSO_4$; since Ba is present, and the substance is entirely soluble in acids, (SO_4) *is absent.*

The following acid-radicles have also been proved to be absent in the general examination for metals :—

$(CrO_4)''$, by the HCl solution not becoming green on passing H_2S.

$(AsO_3)'''$ and $(AsO_4)'''$, by no pp. forming in Group II. B.

$(SiO_3)''$ and $(SiF_6)''$, by no residue insol. in HCl remaining on evaporating to dryness with HCl for Group III. A.

$(PO_4)'''$, by testing with $AmHMoO_4$ in the HCl sol$^{n.}$ for Group III. A.

The organic acid-radicles $\bar{A}$ and $\bar{T}$ are absent, since there was no smell of burning on heating the substance.

The only commonly occurring acid-radicles remaining to be specially tested for are therefore Cl, Br, I, $(C_2O_4)''$, and $(BO_3)'''$; and of these Br and I are probably absent, since no violet fumes of I or brown fumes of Br were evolved with strong H_2SO_4.

Boiled a portion of the substance with pure Na_2CO_3 sol$^{n.}$ and filtered; acidified portions of the filtrate with—

HNO_3	$H\bar{A}$
Added $AgNO_3$, a perfectly white pp., easily sol. in AmHO :— *Pres. of Cl.*	*Added $CaSO_4$*, no pp. :— *Abs. of $(C_2O_4)''$.*

Moistened a piece of turmeric-paper with the HCl sol$^{n.}$ of the substance and dried it at 100°, no reddish-brown stain was produced :—*Abs. of* $(BO_3)'''$.

Acid-radicles found :—CO_3, NO_3, ClO_3, Cl.

Found { *Metallic-radicles :* Cu, Fe, Co, Ba, Sr, Mg, Na, K, NH_4.
Acid-radicles : CO_3, NO_3, ClO_3, Cl.

SECTION VII.

THE LABORATORY AND ITS FITTINGS; APPARATUS, CHEMICALS, AND REAGENTS.

In this Section directions are given which have been found by experience to be of value in fitting and furnishing a laboratory and in maintaining it in working order.

THE LABORATORY BUILDING, AND ITS FITTINGS.

Introductory Remarks.—The Methods of Building, Fitting, Heating, and Ventilating a Chemical Laboratory are of prime importance in securing the comfort, health, and convenience of the laboratory student; in recent years very considerable advances have been made in these matters. For detailed information on these subjects, reference may be made to papers by E. C. Robins, which are published in the *Transactions of the Institute of British Architects* for the years 1882–3 and 1883–4.

1108. **With regard to the Building,** it must suffice to state here that the main points which require attention are that the internal air space, ventilation, and window lighting both from the top and sides, should be secured on as ample a scale as possible. The inner surface of the walls should either be of white glazed brick, which can be easily cleaned when necessary, or the rough brick or plaster surface should be covered with *white* lime-wash or distemper, which can be frequently and cheaply renewed. The windows should be glazed with *colourless* glass.

1109. Draught Closets.—Around the walls, in the spaces between the windows, there should be large and small recesses, each of which is lined with white glazed tiles, has a glazed hood above, and is closed with a sliding sash in front; it is provided at the top with an outlet, through which a strong draught is caused. These draught-closets should be provided with several gas-supplies for Bunsen-burners, the taps being on the outside of the closet; they should also be furnished with a gas-jet for lighting the interior when necessary. The bottom of the closet may be covered with sheet-lead, and should be provided with a small grid through which liquids escape into a drain-pipe. Some at least of these closets should also be provided with a water-supply pipe. A strip of india-rubber draught-excluder fixed along the top of the inner frame and lightly touching the glass, prevents the escape of fumes when the sash is partly opened.

One or more of the larger draught-closets will be reserved for the hydrogen sulphide apparatus (1125). It will be found well to provide such a closet with small doors, which are opened when the gas is to be used: this prevents the gas from escaping in large quantity into the laboratory, as it would do if the large sash were thrown up for each operation. A frame containing pairs of little hinged glazed doors, each about six inches by eight, may be introduced beneath the sliding sash. This enables a student to open as much of the closet-front as is necessary for passing the gas through a solution: while the whole sash can be raised for cleansing or replenishing the hydrogen sulphide apparatus.

One of the draught recesses is kept open for the steam-ovens, small boiler, and condenser which are described in paragraph 1131.

1110. Washing Sinks.—Two or more deep stoneware-sinks should be provided for general use. These have large water-taps fixed above them at some distance from the back of the sink. It will be found convenient if each sink is supplied with three taps. Two of these deliver low-pressure water, hot and cold; and a third is connected with a high-pressure water service, upon which a fire-hose can be at once adapted. A stout wooden board riddled with small perforations may be laid upon the bottom of the sink. It serves as a strainer which prevents solid pieces from getting into the outflow pipe: it also tends to prevent the breakage of glass or porcelain vessels which may be dropped into the sink.

1111. **Working Benches.**—In the frontispiece illustration of this book a block of four laboratory benches in the Nottingham University College is shown. Two of these are presented to the observer, and are therefore visible in detail. This arrangement of the benches is convenient, since it enables the washing-sinks and the water-taps to be placed at the end of each bench and yet to be accessible to each student. The surface of the bench is thus prevented from being wetted and soiled when apparatus is washed.

1112. **Bench Sinks.**—The bench-sinks are of stoneware, with large perforated movable wooden false bottoms, which soften the surface and serve as grids. Half-inch outlet pipes unprotected by metal grids discharge into stoneware pots, the overflow from which into the drain-pipes is through a curved stoneware tube near the top (see Frontispiece). Any fine solid matter, which passes down the sink-pipe, settles in this trap-pot, and is got rid of by taking out the pot and emptying it into a pail at frequent intervals. The main-pipes are occasionally cleansed by flushing them with high-pressure water. Any stoppage in the straight sink-pipe is readily pushed through with a stout cane. Over each sink there are three low-pressure water-taps for washing and for supplying condensers, and two high-pressure water-taps for aspirators.

Lead-covered sinks lessen the risk of breakage arising from the dropping of apparatus while it is being washed, but they are easily corroded by acids and are less cleanly than stoneware. The large wooden grid, which is laid on the bottom of a stoneware sink, is a very efficient and permanent grid, and saves breakages.

1113. **A Stoneware Pocket** is hung upon the end of each working bench to receive waste filter-papers, broken glass, and other solid refuse. This pocket is frequently emptied.

1114. **Bench Draughts.**—Each bench is provided with an aperture leading to a strong draught, and into this aperture a copper hood can be fitted, which serves to carry away acid vapours or badly-smelling gases. The aperture is stopped with a wooden plug when the hood is not in use, in order to improve the draught on other benches. The copper hoods are occasionally cleansed and painted with Brunswick black, in order to protect them against corrosion by acid fumes: fixed light earthenware hoods are fragile, but present the advantage of not being corroded by acids. A glazed draught-hood extending along the top of the centre shelf-rack of the bench has been found to be satisfactory: all heating and evaporation of liquids is then carried out on a raised shelf under this hood.

1115. **The Laboratory Bench** may be made of stout teak or of American walnut-wood, the wood being stopped and varnished with the exception of the working surface of the bench, which should be saturated with oil and then well rubbed. This working surface is preserved by being occasionally thoroughly washed. dried, and rubbed down with a mixture of equal measures of boiled linseed oil and turpentine. Wood of a less durable and expensive kind may be covered with sheet-lead, and will then be found to serve well for the top of the bench.

The bench may be 3 feet in height with a depth of 25 inches; and a space of 7 inches between the reagent shelves above the bench will be found suitable for ordinary bottles. A gangway at least six feet in width should be provided between the rows of benches.

Two gas-taps are fixed on each bench for supplying Bunsen-burners. If gas is not available, Bunsen-burners for consuming methylated spirit or petroleum (1119) may be used with advantage. One lighting-burner is provided at a convenient height for illuminating the bench at night. The incandescent mantle gas-burner is most suitable for the purpose, unless electric glow-lamps are available.

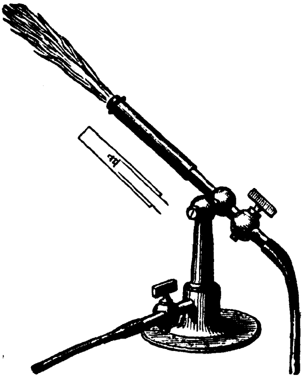
1116. Bench Lockers.—The working-bench is supplied with two lockers, each of which has two drawers above it. In order to lessen the number of locks, which are liable to rust in the laboratory atmosphere, a flat iron bar which swings on a pin is fastened upon a staple by a padlock in front of the two drawers and locker. By this arrangement of lockers two students are enabled to use the same bench at different times. Each locker should have a different key, but all the locks should be under the control of master-keys kept by the attendant and the teachers. The drawers should be about 3 inches in depth, and may be suitably divided by wooden strips. A shelf in the locker should not extend more than half across, so as to leave standing room for tall apparatus.

1117. Preparation Bench.—Besides the ordinary working-benches, there should be at least one long bench, well supplied with gas and water-taps and with overflow-pipes. This bench serves for making up solutions, for distillations, and for other large operations. There should also be a long bench made of a slate slab, and covered at a height of several feet with a metal hood, under which is a good draught outlet. This bench must be supplied with a half-inch gas-pipe and taps, and with water-taps and overflows: it serves to support water-baths, air-baths, combustion furnaces, and gas furnaces of all kinds.

1118. A Blowpipe-table and Blowpipes.—A table, the top of which has a raised edge around it, and which is covered with sheet lead or zinc, should be provided. This should be furnished with gas-taps to supply gas-blowpipes, and luminous-burners for bending glass, and two or more Fletcher-bellows should stand beneath it. It is also well, if possible, to have a water-blowing apparatus near at hand which supplies an air-blast to the blowpipes for long heating operations.

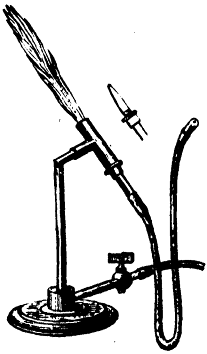
The Fletcher Bunsen-blowpipe (fig. 10, 7) will naturally find its place on this table. It is also well to keep a Bunsen gas-blowpipe (fig. 82) on the table for more elaborate glass-blowing, and a Herapath-blowpipe (fig. 83) for smaller heating operations. The upper part of the Herapath-blowpipe can be purchased without the foot, and this can be fitted into a Bunsen burner.

FIG. 82.



BUNSEN GAS-BLOWPIPE.

FIG. 83.

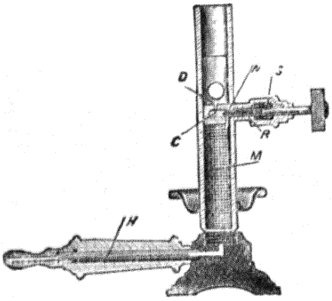


HERAPATH GAS-BLOWPIPE.

1119. Spirit and Petroleum Burners and Blowpipes.— Heating and blowpipe work must occasionally be done without a supply of coal-gas and without an air-blast. Recourse may then be had to the Bunsen-burners and blowpipes which burn the vapour of either methylated spirit or petroleum (figs. 84, 85, 86).

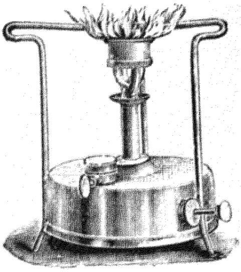
In these apparatus the burner is first heated by burning some methylated spirit from a circular gallery surrounding the burner, and the supply of liquid coming into the base of the burner is vaporised on coming into contact with the hot burner. In the case of the spirit Bunsen (fig. 84) the liquid is supplied to the burner by gravitation from a vessel at higher level. In the petroleum-Bunsen (fig. 85) the petroleum is raised by the pressure of air which is forced by a little pump, shown on the right of the figure, into the upper part of the reservoir. The blast of the blowpipe-lamp (fig. 86) is maintained by the pressure of the vaporised liquid inside the reservoir.

FIG. 84.



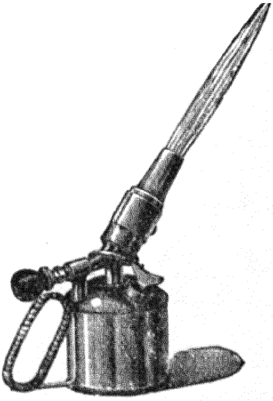
SPIRIT-VAPOUR BUNSEN.

FIG. 85.



PETROLEUM-VAPOUR BUNSEN.

FIG. 86.



BENZOLINE BLAST-LAMP.

1120. Chemical Store-room and Balance-room. — Opening into the general laboratory there should be at least two separate apartments. One of these is fitted with shelves for stores of chemicals and apparatus. The other is reserved for use as a reference-library and for delicate metal apparatus, such as chemical balances, microscopes, spectroscopes, and polariscopes.

The apparatus should stand on a broad shelf which is at a convenient height for sitting; the shelf is supported on iron brackets from the wall so as to secure the chemical balances against disturbance from the vibrations of the floor. The spectroscope and polariscope should have draw-curtains of black lining material, so arranged that when the instruments are in use, light from outside may be excluded

A large central table should also be provided in the balance-room for use in writing and reading. A special low table, with a raised flange, for gas-analysis apparatus and for other experiments involving the use of mercury, is necessary: and in order to provide against the loss of mercury spilt from these tables, it will be well to have the floor covered with linoleum with turned up edges.

BENCH-APPARATUS.

Detailed descriptions of this apparatus are rendered unnecessary by the existence of fully illustrated and descriptive price lists, which are now furnished by the apparatus-sellers.

1121. A list of the apparatus which should be kept in each bench-locker will be found on page xxiii. The separate items of that list are repeated here with the addition of details of dimensions and of other useful information.

In Section I. of the book, directions are given for preparing and fitting this apparatus.

The Bunsen-burner with its india-rubber tube may be left attached to the gas-tap on the bench; all the other apparatus should be locked up in the bench-locker when it is not in use. Most of this apparatus is seen on the bench in the Frontispiece illustration.

1 Bunsen-burner (1), about 5½ inches in height, with ⅜-inch tube and with means of closing the air-holes.
1 Rose-top to fit the burner (1).
1 Fletcher's Argand-Bunsen burner (2), ¾-inch size, is convenient for boiling the wash-bottle.
1 Piece of red or black india-rubber tubing, $\frac{5}{16}$-inch in internal diameter and 16 inches long, to supply gas to the burner.

1 Test-tube stand with twelve holes, two of which are at least 1 inch across ; best without draining-pegs.
1 Test-tube brush (13).
12 Test-tubes, 5 inches long, ⅝-inch in internal diameter.
2 Boiling-tubes, 6 inches long, 1 inch in internal diameter.
2 Round glass plates, ground on one side, 3 inches across.
2 Berlin-porcelain evaporating-dishes with spouts, glazed inside and out, and 3 inches in diameter.
2 Watch-glasses, 2 inches across.
1 Conical flask of 4 ounces capacity (fig. 33, p. 43).
1 Wedgwood-mortar, 4 inches across, and pestle with wooden handle.
1 Galvanised iron tripod-stand, 7 inches in height, with round top 4 inches across (fig. 33, p. 43) ; these dimensions are suitable to the above Bunsen-burner.
1 Square of coarse iron wire-gauze, 5 inches in the side ; best with the corners clipped off (fig. 33, p. 43.)
(A square of asbestos millboard may be used instead of the gauze.)
3 Plain glass funnels, two of them 2½ inches across, and one 2 inches across.
3 Beakers without lips, wide form, of 1, 2, and 3 ounces capacity.
3 Glass rods, round at the ends, 7, 6, and 3 inches in length (10).
1 Piece of platinum foil, 1 inch by 1½ inches.
2 Pieces of mounted platinum wire (9), each 2 inches long and about as stout as an ordinary sewing needle.
1 Blowpipe, Black's japanned tin (fig. 9, p 6).
1 Pipe-clay triangle, 2 inches along its side.
1 Wash-bottle (12), made by fitting a conical 18-ounce flask, which is at least 1 inch across the inside of the neck.
1 Retort-stand of galvanised iron (fig. 33, p. 43), upright rod 16 inches, foot 6 by 3½ inches, with 3 rings of brass or gun-metal, the largest 3 inches across.
1 Wooden filter-stand (fig. 49, p 71), rod 12 inches high, two rings on one boss, each 2¼ inches across, foot 5 by 8 inches by ½ inch.
1 Pair of polished brass crucible-tongs, 6 inches long.
1 Small horn spatula, 3½ inches long.
1 Wicker oval draining-basket, 10 inches by 8, and 4 inches deep.
Cut filter-papers, 4½, 3½, and 2¾ inches across.

SPECIAL APPARATUS FOR SECTION II.

1122. The following Apparatus is required for the experiments on gases; it need not be supplied to each student, but several sets should be kept in accessible positions in the laboratory, whence they may be fetched when they are required. This apparatus should never be kept in the bench-lockers. An asterisk indicates that the apparatus is also required for chemical analysis.

*1 Nest of three or four of the smallest sizes of brass cork-borers.
*1 Triangular file, without wooden handle.
*1 Thin round file, without wooden handle.
* Several lengths and pieces of hard glass tubing, about $\frac{1}{8}$ inch internal diameter.
* Ignition-tubes, 3 inches long by $\frac{1}{2}$ inch across, and 3 by $\frac{3}{16}$.
* Corks as free as possible from holes and cracks, sizes varying from $\frac{3}{8}$ to $\frac{3}{4}$ inch across.
1 Small metal clamp, in a boss which fits on the retort-stand (see fig. 21, 23).
1 Stoppered bell-jar of 16 ounces capacity.
4 Glass cylinders on feet (fig. 22, 24), with ground edge at top, 8 inches high, $1\frac{3}{4}$ inches across.
4 Cylinders similar to the above, 6 inches high, $1\frac{1}{2}$ inches across.
1 Round brown stoneware trough, 12 inches across, 5 inches deep.
1 Metal deflagrating-spoon, with brass cap $2\frac{1}{2}$ inches across.
2 Two-necked Woulffe's bottles of 8 ounces capacity.
2 Two-necked Woulffe's bottles of 4 ounces capacity.
1 Tubulated retort of 6 ounces capacity.
2 Thistle-funnels, 8 inches long.
1 Clock-glass, about 4 inches across.
4 Pieces of red or black india-rubber tubing, $\frac{3}{16}$ inch in internal diameter and $1\frac{1}{2}$ inches long.
* A good supply of wooden spills, or thin strips of wood.
* A good supply of wax tapers, about 11 inches long.
* A small piece of thin candle.
1 Tube (fig. 26 *a*, 33), 6 inches long, filled with small pieces of calcium chloride.
4 Strips of sheet-lead, 6 inches long, $\frac{1}{2}$ inch broad.

APPARATUS FOR GENERAL USE IN ANALYSIS.

1123. The Following Apparatus should be kept in the laboratory for the general use of students who are doing chemical analysis.

One set will suffice for about twelve students. Each article should have its own place in the laboratory, and should be replaced immediately after use, since it may be required by other students. The apparatus mentioned in paragraph **1122**, which is marked with an asterisk, is also required for analysis, and must be included in the following list:—

1 Spirit-lamp, 4 ounces in capacity, with earthenware wick-holder and ground glass cap.
1 Charcoal borer of conical form.
4 Tin filter-dryers.
4 Berlin-porcelain crucibles, 1½ inches across, with covers.
1 Iron mortar, 8 inches across, with pestle.
1 Fletcher's No. 5 foot-bellows (fig. 11, 7), and a blowpipe-table covered with sheet zinc or lead.
1 Fletcher's Bunsen-burner blowpipe, with central blast, and two taps (fig. 10, 7).
1 Bunsen-blowpipe, and one Herapath-blowpipe (1118).
1 Fletcher's solid-flame burner, with tripod for large dishes (fig. 3, 2).
1 Fletcher's injector-furnace for strongly heating large crucibles.
1 Microscope, with at least a ¼-inch object glass.
1 Spectroscope, Bunsen's table form (115)
1 Spectroscope, direct vision, pocket form (114).
1 Indigo-prism, stoppered.

This prism is nearly filled with solution of indigo in strong sulphuric acid; the dry stopper is inserted into the dry neck and is securely fastened down with fine copper wire; melted paraffin wax is then allowed to flow over the stopper and neck.

The indigo-solution is made by mixing commercial sulphindigotic acid with about ten times its measure of strong sulphuric acid, letting settle for several days, and decanting into the prism.

1 Agate mortar 3 inches across, and pestle.

This mortar is used for reducing hard substances to fine powder, after they have been already broken into minute fragments. The process of powdering in the agate mortar must be effected by trituration; blows must on no account be struck with the pestle, else the mortar may be broken.

2 Small leaden cups, about 1½ inches across and ¾ inch deep (606).

These are made by beating sheet-lead into shape.

2 Copper water-baths, 5 inches across, and with four diminishing rings (88).

One large bath with several openings (88) may replace these smaller baths. The baths should be about two-thirds filled with water, and the flame should be so adjusted that the water is kept just boiling. Evaporating basins placed on the bath cannot be heated above 100° C.

2 Pairs of conical flasks, fitted as is shown in fig. 70, 315.
2 Tubulated flasks, 4 ounces in capacity (fig 73, 505).
2 Clarke's retorts with condensers (fig 74, 555).

A box of assorted pieces of wood charcoal, as free as possible from cracks and from bark.

HYDROGEN SULPHIDE APPARATUS.

1124. **Hydrogen sulphide Gas** is constantly required by the analyst, and should be readily obtainable at any time in a constant stream. Arrangements must be made for preventing this badly-smelling gas from polluting the atmosphere of the laboratory to any large extent. Both these objects, together with economy of materials, are better secured by the use of a large apparatus in common by many students, than by the employment of a small private apparatus by each student.

The gas is most readily obtained by the action of somewhat diluted cold commercial hydrochloric acid upon fragments of ferrous sulphide.

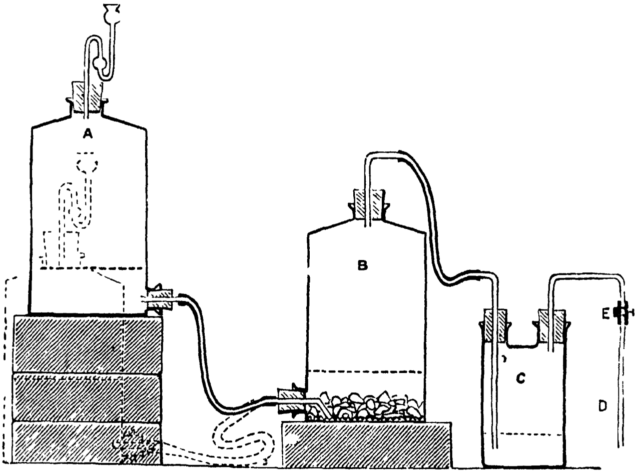
1125. **General Hydrogen sulphide Apparatus.**—The apparatus in which the gas is prepared has assumed many forms. The one shown in section in figure 87 and described below, is recommended by its simplicity, efficiency, and cheapness.

Two large tubulated bottles, A and B, of at least two quarts capacity each, are fitted as is shown in the figure. The tubulures of the bottles are connected together by a piece of broad rubber tubing, and the bottle B is also connected with the wash-bottle C by narrow rubber tubing. Rubber stoppers should be used, since they are more durable than ordinary corks and are always gas-tight. The delivery tube D is connected with the doubly-bent tube from the washing-bottle by means of a rubber joint, which can be partially or entirely closed by means of a screw-clamp E.

In charging the apparatus, the bottle B is laid on its side and is then filled with lumps of ferrous sulphide. The washing-bottle C is then partly filled with water, and the three bottles are connected. The clamp E is now closed, and the bottle A is about half filled with a mixture in equal measures of strong commercial hydrochloric acid and water.

When the gas is required, the bottle A is raised upon a suitable stand, and the clamp E is gradually opened until a regular stream of bubbles passes through the water in C.

FIG. 87.



LARGE HYDROGEN SULPHIDE APPARATUS.

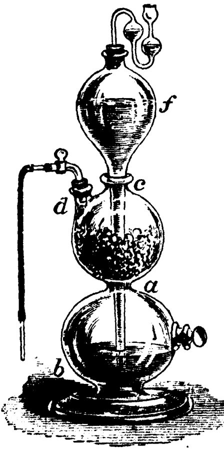
A part of the acid will flow from the bottle A into the bottle B, and will cause hydrogen sulphide to be evolved from the ferrous sulphide; the gas will be forced out of B by the pressure of the acid which remains in A. Before it escapes through the tube D, the gas is washed free from small drops of liquid, containing HCl and $FeCl_2$, by passing through the water in C. The levels of the liquids in the bottles A, B and C, while the gas is being produced, are shown by the horizontal dotted lines.

When the gas is no longer required, the screw-clamp at E is gradually tightened until the stream of bubbles through the water in C is arrested. The gas then accumulates in B, and forces the acid out of B back into A. If the glass tube at the bottom of B is bent, as is shown in the figure, the acid will be almost completely expelled. This will cause the production of the gas to cease. The evolution of gas will recommence, however, as soon as the clamp E is opened.

During the hours of laboratory work A should remain raised on its stand, and the apparatus will then always be ready for use. At other times A should be lowered into the position represented in dotted outline in the figure. B should be permanently somewhat raised in order to facilitate the outflow of the acid when A is lowered. The escape of the hydrogen sulphide gas from solution in the acid is much lessened in amount, if a funnel containing water or glycerine is fitted into the stopper at the top of A, or if a loosely-fitting glass stopper is dropped into the neck of the bottle.

When the acid is spent and no longer causes evolution of gas from the FeS, it is poured away and replaced by fresh acid. Lumps of FeS are also occasionally placed in B, as the substance becomes dissolved.

FIG. 88.



THE KIPP-APPARATUS.

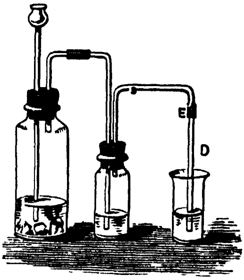
The Kipp-apparatus (fig. 88) may also be used for generating hydrogen sulphide. It is similar in its action to the one already described, but the acid vessel (*f*) is placed above the generating vessel (*c*), and the two are connected together rigidly when the apparatus is fitted up for use.

The hydrogen sulphide apparatus should stand in a closet, which is lined with glazed white tiles and is furnished with a good draught. The bottom of the closet should be covered with sheet-lead, and furnished with a drain-pipe for the spent acid, and a gas-jet should be provided for lighting it at night. The closet must not be used for other purposes.

A large opening in the closet should be arranged for recharging the apparatus; but a small door only should be used for introducing the solutions through which the gas has to be passed. If the front of the closet is opened by means of a glazed sliding-sash, a frame containing pairs of glazed hinged doors, each about six inches by eight, may be introduced into the grooves beneath the sash. These doors enable the apparatus to be used with the least possible opening of the closet. Such an apparatus should serve for the use of twelve or more students, according to the character of the work which they are doing.

1126. **An Apparatus for Private Use** may be fitted as is shown in figure 89. The larger bottle contains pieces of ferrous sulphide, upon which HCl, diluted with an equal measure of water, is poured through the thistle-funnel. The gas passes through a little water in the smaller bottle, and thence into the solution to be saturated. The use of a small apparatus by each student in a large laboratory is not to be recommended, since it causes unnecessary waste of time and materials, and does not permit of satisfactory control of the stream of gas which is evolved.

FIG. 89.



SMALL HYDROGEN SULPHIDE APPARATUS.

1127. Passing Hydrogen sulphide.—Each student should keep a glass tube D (figs. 87, 88, 89), which may be fitted upon the hydrogen sulphide apparatus in order to conduct the gas into a solution. When a liquid is to be saturated with gas, the end of this tube should be passed down *to the bottom* of the vessel which contains the liquid. A regular stream of gas is then caused to bubble through the liquid by gradually opening the clamp E (fig. 87), or the stopcock *d* (fig. 88) of the larger apparatus, or by introducing a sufficient quantity of acid into the larger bottle of the small apparatus (fig. 89).

In order to avoid unnecessary waste of gas, the liquid should be of sufficient depth to absorb much of the gas, and the bubbles should not pass too fast to be readily counted. The passage of the gas should be continued for four or five minutes if the quantity of metal to be precipitated is large, and the liquid must smell strongly of the gas after the air above it has been blown out and the liquid has been shaken. The tube D must be thoroughly cleansed immediately after use.

Liquid Hydrogen sulphide can now be purchased in metal bottles. When the valve of the bottle is slightly opened, a stream of the pure gas will escape. A store of ready-made hydrogen sulphide may be kept in this form, and the trouble of maintaining the apparatus for preparing the gas may thus be obviated.

1128. Hydrogen sulphide Solution.—It will be found convenient to keep a bottle of distilled water saturated with hydrogen sulphide. This solution is prepared by passing a regular stream of the gas through water which nearly fills the bottle. On removing the bottle, closing its mouth tightly with the thumb and shaking its contents violently, no suction must be felt on the thumb, but a slight outward pressure: this proves that the water is saturated with the gas.

DISTILLATION OF WATER.

1129. As has been already stated (82), water ordinarily contains certain solid substances in solution, which render it in a chemical sense impure. Such water is therefore unfit to be employed for the processes of solution and of washing precipitates, since any impurity thus introduced into a substance during analysis, would be considered, when detected, to have been present in the original substance.

The quantity of these impurities which is present in any particular water-supply will depend upon the nature of the soil with which the water has been in contact before its collection. The water-supply in some districts will be found to be almost perfectly pure, when it is subjected to the tests for impurity (1162, Remark 41). Rain-water which has fallen through pure air, and has been carefully collected, will yield little or no indication of dissolved impurity. The water-supply to the majority of laboratories will, however, be found to be unfit for analytical purposes, until it has been freed from dissolved solid substances by being subjected to the process of distillation.

In most towns distilled water can be purchased ; but it is preferable to distil all the water required for use, either in the laboratory itself or in its immediate neighbourhood. For this purpose steam is condensed by a block-tin worm-pipe, which is immersed in a tub through which a constant stream of cold water runs. The steam may be obtained either from a steam-boiler, or from a copper still heated by a furnace or gas-burner, or from the water-baths and steam-ovens in the laboratory, as is described below.

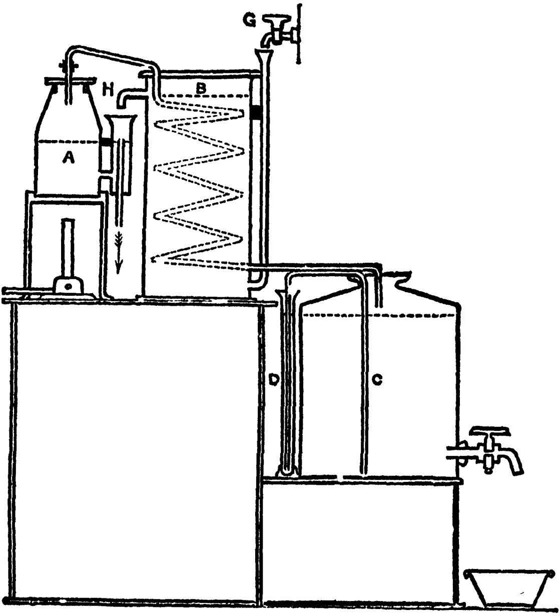
1130. **The Process of Distillation** may be carried on continuously in the constant still which is figured and described below. The sketch in figure 90 renders a lengthened description of this still unnecessary. It represents the apparatus in section and about one-twentieth its working size; the side-feed to the boiler is about one-tenth its usual breadth.

The water in the copper still (A) is heated by the flame of a Fletcher-burner or of a large-sized Bunsen-burner. The steam thus generated passes from the top of the still through a spiral tin tube immersed in water in the condensing-tub (B), which is made of copper or of galvanised iron From this condenser the distilled water flows into the large stoneware vessel (C), which is furnished with a tap below from which the water may be drawn when required. The long syphon tube (CD) serves as a gauge to indicate the level of the water inside the jar.

The condensing-tub (B) is constantly supplied with cold water from a tap (G); this water flows through the funnel tube to the bottom of the condenser. The overflow of hot water passes from the tube (H) at the top of the condenser into a side-feed for supplying the boiler. From the side-feed the excess of water flows through a central tube to a sink, as is shown by the arrow.

The spiral block-tin pipe in the condenser must be of sufficient diameter to prevent it from offering any obstruction to the free escape of the steam from the boiler. Every part of this tube must have a downward slope so as to prevent water from collecting in it, and the coils must be supported in position by a suitable framework of wood or metal, which prevents them from sinking.

FIG. 90.



AUTOMATIC WATER-STILL.

The copper still should be furnished with a broad lid screwing upon its mouth, for convenience in removing the deposit which collects within. Into this lid a brass tube should be fastened which carries a screw-union. This union enables the tin condensing-pipe to be connected steam-tight with the still.

By the above arrangement the still is constantly supplied with the hottest water from the condenser through the side-feed, and the water in the still is always maintained at the level shown by the dotted line.

When the apparatus has been properly fitted up and the supply of water has been duly adjusted, it is only necessary to light and extinguish the gas under the still when the distillation is started and stopped. It is advisable to remove the incrustation occasionally from the interior of the still, and from the supply-tube which connects the still with the condensing vessel, by means of diluted hydrochloric acid.

The wooden stands which support the still and the storing-jar may be conveniently fitted with shelves and doors so as to serve as store-closets.

When such a copper still, measuring 23 inches in circumference below and 8 inches in height, is filled to a height of 4 inches and is heated by the largest Bunsen-burner, it will yield about two litres of distilled water per hour.

In laboratories which require a larger supply of distilled water than can be furnished by the above apparatus, a copper still of several gallons capacity may be set in masonry and heated by a small furnace fed with coal, coke, or gas. The overflow from the still-tub should then pass into a small cistern, the overflow-pipe of which keeps its water-surface level with that required in the still. The still is supplied with hot water by connecting it with this cistern by means of a tube, which is bent downwards in its middle so as to hinder the circulation of water between the still and the cistern.

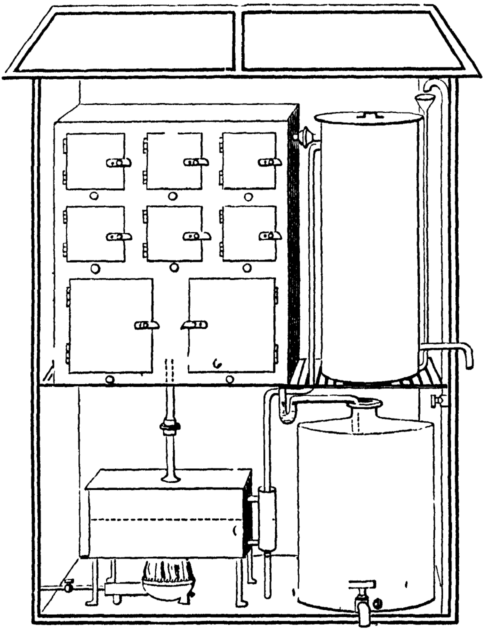
DISTILLATION OF WATER THROUGH STEAM-OVENS.

1131. **The Distillation of Water may be combined with the Heating of Drying-ovens** to a temperature not exceeding 100° C. For this purpose one of the tiled recesses in the laboratory wall is furnished with a glazed projecting hood and with a good draught from the top. Into this recess the arrangement represented in figure 91 is fitted. It is drawn about one-sixteenth its full size.

A copper boiler and a stoneware collecting jar are seen resting on the floor of the recess, while the set of double-cased copper-ovens and the still-tub are supported on a strong iron grating above.

The steam is generated by a powerful Fletcher-burner, and passes from the boiler through a pipe which terminates several inches above the bottom of the outer casing of the ovens. It then makes its way between and around the various ovens, and undergoes partial condensation in heating them. The hot condensed water, thus produced, flows through a pipe, the opening of which is flush with the bottom of the oven casing, into the stoneware jar. From this jar hot distilled water may be drawn when the apparatus is at work. The pipe from the bottom of the oven has a U-bend which prevents the escape of steam.

FIG. 91.



AUTOMATIC WATER-STILL AND STEAM-OVENS.

Any steam, which is not condensed in heating the ovens, passes from the upper part of the oven-casing into a block-tin worm inclosed in the tall still-tub. It is thus condensed, and drops as distilled water from the end of the worm-pipe into a second stoneware store-jar which is provided with a tap below. The level of the water inside this jar is shown by a syphon-gauge, such as that seen in figure 90. The coils of the spiral tin pipe must in all parts have a downward slope so as to cause the water to flow away rapidly; these coils must be supported in the condensing-tub by a suitable frame in order to prevent them from bending down out of position.

The still-tub is supplied with a stream of cold water from a pipe, in which is a control tap seen at the right-hand side of the figure. From the opposite side of the still-tub the heated condensing-water flows into a little feeding vessel attached to the side of the boiler: this is shown in section in figure 93. A portion of this hot water serves to keep up the water-level in the boiler to the dotted line shown in the figure, and the rest flows away into the drain-pipes through a central pipe which is seen in the feeding vessel (fig. 93).

All the connections are made by means of screw-union joints; the apparatus can, therefore, be quickly and easily disconnected for removing the incrustation in the boiler and on the condensing worm and for repairs.

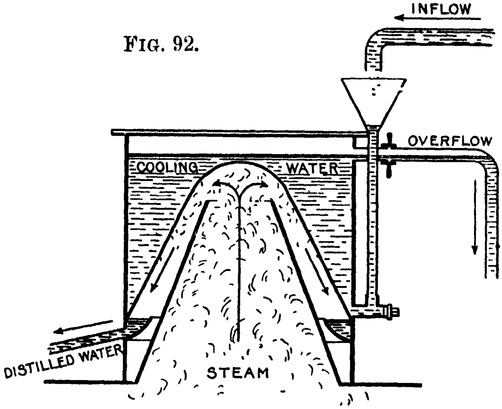
This apparatus may be allowed to run uninterruptedly for many days and nights in succession without any attention, after the gas burner has been lighted and the supply of condensing water has been regulated by the tap. It has for many years kept a large students' laboratory supplied with distilled water, and has also furnished the necessary steam to the drying-ovens.

It has been found possible to attain a higher temperature than that usually recorded in a steam-oven, by dispensing with the usual inlet for air through the door. The air is admitted through a narrow copper tube, one end of which is seen immediately beneath the door of the oven, and the other end opens flush into the middle of the bottom of the oven. The pipe itself is thus constantly surrounded with steam, and the air which passes through it into the oven enters at a temperature of nearly 100°. A small outlet for air is provided at the upper part of the back of each oven.

It has also been found convenient to arrange that the doors of the ovens shall consist of metal frames in which a sheet of plate glass is fastened : this renders the contents of the oven visible without opening the door.

An Improved Form of this apparatus, known as the Brown-still, has the bottom of the ovens converted into a shallow boiler, which is heated by a long Fletcher-burner. The condenser is mounted on the top of the ovens and has a domed copper surface for condensation, with a circular collecting trough at its bottom, as is shown in the sectional drawing (fig. 92).

FIG. 92.



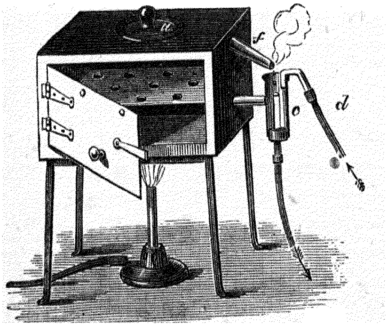
THE BROWN-CONDENSER.

THE STEAM-OVEN

1132. The Steam-oven is a small oven of sheet copper, which is used for drying substances at a temperature not exceeding 100° C. (fig. 93, and fig. 57, 98). These ovens may be heated by steam from a separate boiler (fig. 91), and this method prevents the oven from being coated inside with incrustation and from being burnt by the heating flame ; but the oven is often heated by water which is kept boiling in the space between the oven itself and the copper casing which surrounds it (fig. 93). The outer casing may have a circular opening cut in the top, which serves as a water-bath : this is covered by a lid when it is not in use (fig. 93 *a*).

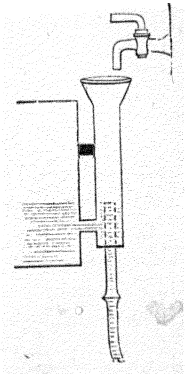
The level of water in the jacket may be maintained constant by a contrivance which is shown in section at *c* (fig. 93) and by a special sectional drawing in figure 94. A small reservoir communicates by a lateral tube with the space between the oven and its jacket, and is constantly fed with water from a supply-tube (*d*) or from a water-tap. The overflow of this reservoir is a central tube, the end of which terminates above the level of the lateral tube. The outlet for steam (*f*, fig. 93), may be turned downwards, so that any water which is formed by the condensation of the steam drops into the reservoir (*c*); or it may be made to communicate with the worm of a condenser, when the steam will be condensed into distilled water.

FIG. 93.



WATER-OVEN.

FIG. 94.



AUTOMATIC WATER-FEED.

The steam-oven should if possible be supplied with soft water or with distilled water; the formation of a troublesome incrustation in the interior is thus prevented. A raised vessel of distilled water may be connected with the supply-pipe (*d*), and the overflow may then be collected and occasionally returned to this supply vessel.

Or better still, the escape-pipe (*f*) may be turned upwards and connected with a long vertical or oblique tube, which will condense the escaping steam and return it to the oven. This arrangement obviates the necessity of a constant supply of water. In this case the water in the oven should not be allowed to boil too vigorously, else the steam will not be completely condensed by the vertical tube.

RECOVERY OF SILVER AND PLATINUM FROM THEIR RESIDUES.

1133. Waste scraps of platinum and silver, and solutions and precipitates which contain these metals, should not be thrown away, but should be preserved in two large jars which are specially reserved for them, and are labelled *Silver Residues* and *Platinum Residues* respectively. Platinum chloride and silver nitrate may be obtained from these residues by the methods described below.

SILVER RESIDUES.

1134 Some HCl is poured into the jar which contains the silver residues, and the acid is thoroughly mixed with the contents of the jar. The precipitate is then allowed to subside, and, after the liquid has been decanted, this precipitate is thoroughly washed by decantation: it may then be treated either by paragraph **1135** or **1136**.

1135. The wet precipitate is rinsed into a flat shallow dish, and the layer of water is acidified with H_2SO_4. Strips of Zn free from Pb are then laid upon the silver precipitate, and the whole is allowed to stand quietly for several hours. The black spongy mass which remains is metallic silver. This is separated from any remaining Zn, and is then washed until it is free from H_2SO_4.

This metallic deposit is dissolved by heating it with pure strong HNO_3, which has been diluted with its own measure of water. The solution is evaporated to dryness; the solid residue is dissolved in water, and is once more evaporated to dryness in order to remove free acid completely. The residue is then dissolved in the proper proportion of water to give reagent 48 (**1161**). Any residue insoluble in HNO_3 is returned to the silver residue bottle.

1136. The well-washed precipitate which has been obtained from the silver residue bottle, after the addition of HCl in excess (**1134**), is filtered off and dried. It is then mixed with twice its weight of a mixture in equal proportions of K_2CO_3 and Na_2CO_3. This mixture is transferred to a clay crucible and is fused in a furnace. After the contents have been kept in a fused condition for four or five minutes, the crucible is removed and its bottom is tapped several times on a brick so as to cause the melted globules of Ag to unite. When the crucible is cool it is broken up, and the button of Ag is removed and well washed. It is then converted into $AgNO_3$-solution, as is described in paragraph **1135**.

PLATINUM RESIDUES.

1137. The liquid in the Platinum residue bottle is shaken up with the precipitate, and the whole is evaporated to dryness in a porcelain dish. The residue is then heated strongly for some time. When the dish is cool, water is poured upon the residue and boiled with it, and is then decanted off. Solution of oxalic acid is now poured into the dish and evaporated to dryness, and the residue is once more ignited strongly.

The residue of metallic Pt is thoroughly washed with boiling water, and is dissolved by heating it with HCl, to which one-third its measure of HNO_3 has been added. The solution is evaporated to dryness over a water-bath. HCl is then poured in, and the liquid is once more evaporated to dryness and heated for some time on the water-bath. This residue, when dissolved in water, forms reagent 28 (**1161**).

Waste scraps of platinum foil and wire should be carefully preserved. They are cleansed by boiling them with HNO_3 and washing them well with water. They are then dissolved in a mixture of HCl and HNO_3, and converted into solution of $PtCl_4$, as is described in the preceding paragraph.

CHEMICALS AND REAGENTS.

In the Following Paragraphs general Directions are given for the preparation of the reagents and of the test-substances which are required in the preceding Analytical Course.

Tabulated lists of the names and formulæ of these substances follow the general directions. A reference number is attached to each substance, and certain necessary details concerning its preparation and the tests for its purity are also supplied. Lists of the chemicals which are required for the experiments in Sections I. and II., and of certain special reagents for detecting the Rarer Elements and Organic Substances, are also given.

The pure chemicals which are required in analysis are readily purchased, and it will be found more economical as a rule to buy them than to prepare them.

The preparation of the solutions, and the dilution of acids and other liquids, should, however, always be performed in the laboratory, since this lessens the cost both of purchase and of carriage.

Substances suitable for analysis are suggested in paragraphs 1168–1172.

1138. **Solution and Dilution.**—Many reagents and test-substances must be dissolved or diluted before they are used. As these processes of solution and dilution must be constantly carried out in a laboratory, it is important that the methods employed should be as simple and rapid in execution as possible, in order that the expenditure of labour and of time should be minimised.

It is also of great importance that the solutions should be of appropriate strength. It is usually advisable that the same liquid should be of different strengths when it is used as a reagent and as a test-solution. In either case a strength can be selected which generally yields the most satisfactory results. If the solution is stronger than this, chemicals are wasted; if it is weaker than this, the reaction is not satisfactorily obtained. Hence it is advisable to keep these liquids in readiness for the student, rather than to leave him to prepare them doubtfully or wastefully when they are required.

In order that the labour of preparing these solutions may be reduced to a minimum, it is well to make each solution in some quantity and to keep it in stock.

1139. The Apparatus required for Preparing Solutions includes—

A common pair of scales, with earthenware slabs and beam below
A more delicate pair of ordinary scales.
A set of brass weights from 1 kilogram to 1 gram.
A strong glass measuring-cylinder to deliver 1 litre, and with graduations for every 5 or 10 cubic centimetres (c.c.).
Several large wide-mouthed gallon jars, some of stoneware or earthenware, others of common green glass.
Several large funnels of glass or of Wedgwood ware.

A convenient Store-bottle for Liquids is a well-cleansed *Winchester-quart*, the bottle in which acids and other liquids are constantly supplied to the laboratory.

The process of preparing liquid reagents is naturally divided into the *Dilution of Liquids* (1140), the *Solution of Solids* (1141–1146), and the *Solution of Gases* (1147–1153).

DILUTION OF LIQUIDS.

1140. In the Lists of Diluted Liquids which Follow, the proportions by measure, in which the liquids are to be mixed with distilled water, are stated. Liquids may be mixed in these proportions by measuring them before they are mixed. But the process is often simplified by measuring the height from the bottom of the cylindrical vessel in which the mixture is to be made, to the level which the mixture is to reach. This height is then divided in the requisite proportion; and the division is marked by a file or a diamond, by a painted line, or by an India-rubber ring. Each liquid is then poured in to its own level, and the liquids are thoroughly mixed by shaking the bottle. The mark, when once made, will always serve for diluting the same liquid again in the same vessel.

Thus dilute HCl, No. 2 (**1160**), is made by mixing strong HCl with three times its measure of water. The height of a Winchester-quart bottle from its bottom to its shoulder was found to be 8 inches. A mark was therefore made on the bottle 2 inches from the bottom, and strong acid was poured in until it reached this mark. The bottle was then filled to the shoulder with distilled water, and stoppered and shaken. It was thus quickly filled with dilute acid of the required strength.

This method serves for numbers 2, 3, 4, 6, and 7 (**1160**).

In the preparation of dilute H_2SO_4 (1, **1160**) special precautions are necessary, because the strong acid becomes heated by dilution, see Remark 1 (**1160**).

SOLUTION OF SOLIDS.

1141. In the Lists of Dissolved Solids which Follow, the proportions of solid to water, which are to be used in preparing each solution, are always placed in the Fourth and Fifth Columns.

In some of the Tables the Fourth Column gives the number of grammes of the solid to be dissolved in the Winchester quart of water. This number is derived from the proportion of solid to water, by considering that when an average Winchester-quart bottle is filled about an inch above its shoulder, it holds 2400 c.c. (cubic centimetres), or approximately 2400 grams, of distilled water.

In a similar manner the weight of water held by any other bottle or vessel may be obtained with sufficient accuracy for this purpose by measuring its capacity in c.c., and counting this number as the weight of water in grams.

Thus the strength given for the AmCl reagent-solution, No. 5 (**1160**), is 1 : 8, as is shown in the Fifth Column. This means that a given weight of water must be made to dissolve one-eighth its weight of solid AmCl. Since a Winchester-quart bottle holds 2400 grammes of water, one-eighth of 2400, or 300 grammes, of AmCl must be dissolved in this water in order to obtain a solution of the required strength.

Pure solid chemicals are usually sold in the crystalline condition. Hence it may be assumed that the crystalline substance is to be used, unless (*a*), which signifies amorphous, is placed against the name in the first column in the table.

1142. If a Small quantity of a Finely-powdered Solid has to be dissolved, or if the solid is extremely soluble in water, the solution is often most simply made by introducing the weighed solid at once into the water contained in a stoppered bottle or beaker, and then shaking or stirring the water until solution is completed. This method is successful with No. 10 (**1160**), with Nos. 28, 34, 38, 48, 53, 61, 63, 70, 77, 78, 81 (**1161, 1162**), and with many of the substances in paragraphs **1163, 1164.**

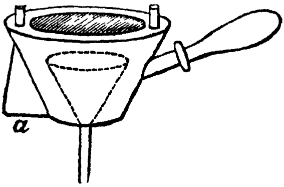
1143. For Dissolving Large Quantities of Solids two methods are described below. The first (**1144**) is useful when the solution must be rapidly prepared, and is of general application to the solution of solids; but the second (**1145**) is decidedly preferable for many reasons, and should always be employed if possible.

1144. First Method.—Weigh out the required quantity of the solid substance, and heat it with distilled water in a large porcelain dish over a Bunsen-burner with a rose top, or over a Fletcher-burner.

This solution would often crystallise as it cooled; hence, if it is not clear, it should either be filtered in a hot filter-jacket (see *Note*, below), or it may be at once poured into some cold distilled water contained in a large beaker, which has been marked to show the quantity of liquid required to fill the store-bottle. While the liquid is being poured out of the dish, a wet glass rod should be held vertically and pressed against the lip of the dish, and the bottom of the dish should touch the top of the beaker. These precautions will prevent the liquid from running down outside the beaker.

Note.—**The Filter-Jacket** (fig. 95) is a double-walled conical vessel, which is shaped to fit the funnel, and which has a hollow projection (*a*) communicating with the interior of the jacket. Before the apparatus is used, it is about half-filled with water. The water is then heated to boiling by placing a Bunsen-flame beneath the projection (*a*), which is filled with water communicating with that in the jacket. In this way hot water will circulate throughout the copper jacket, and thus the funnel supported in it may be kept hot during filtration.

FIG. 95.



FILTER-JACKET.

The solution is now diluted with distilled water to the required volume, if this has not been done already: the liquids are mixed well by stirring; and if the solution is turbid, it is covered and allowed to stand until it is clear. It is then poured off into the store-bottle, the sediment being left behind.

If the liquid is wanted in haste, it may be filtered into the bottle. For this purpose a double filter or a fluted filter may be used. If the liquid is filtered before it has been diluted to the full extent, the quantity to be passed through the filter will be lessened and time will be economised.

The Operation of Filtration, as it is described for ordinary analytical purposes in paragraph 92, is very slow when a large quantity of a solid has to be separated from a liquid. This is partly due to the fineness of the pores of the filter-paper, and partly to the fact that the paper is in close contact with the sides of the funnel.

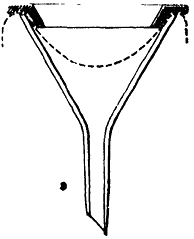
Methods are described below which serve better for dealing with large quantities of material to be filtered, such as are used in the preparation of pure chemicals and of stock solutions. It will be noticed that the method to be employed varies with the character of the solid.

Gelatinous or Non-granular Solids are conveniently separated from the liquids which contain them, by filtration through a large surface of fine linen.

One method for effecting this is shown in figure 96. The linen is first washed in hot water until it is free from glaze it is then placed in a large funnel and made to assume the shape of a bag, as is shown by the dotted line. The bag is kept in its place by pressing a ring of wood firmly into the top of the funnel, as is shown in shaded section When the filtration is finished, the bag may be squeezed so as to remove the adherent liquid more completely from the solid

A simple modification of this method consists in making a square wooden frame about six inches across, and driving in a wire nail at each of its corners. The heads of the nails are then cut off so as to form four pins upon which the linen can be hung in the form of a bag. The funnel is thus dispensed with.

FIG. 96.



LINEN FILTER.

Granular or Crystalline Solids may be conveniently separated from the liquids which contain them, by means of a circular perforated porcelain plate about two inches in diameter.

The perforated plate is dropped into the funnel and remains about 1¾ inches from the apex of its conical interior surface.

The plate is then covered with a disc of filter-paper or of linen, which should be very slightly larger than the plate. After the moistened filter has been pressed into close contact with the inside of the funnel, the liquid which is to be filtered is poured upon the filter. Filtration will take place much more rapidly in this filter than in an ordinary paper filter which is in contact with the funnel by its whole surface.

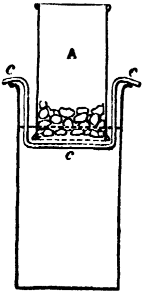
1145. THE SECOND METHOD depends upon the fact that the specific gravity of water becomes higher as the quantity of solid dissolved in it is increased. Hence if the solid is immersed just beneath the surface of the water, a circulation of the liquid is produced. The water which has been in contact with the solid, and has been increased in specific gravity by dissolving it, constantly sinks and is replaced by the comparatively lighter liquid from below. This circulation of water over the solid substance gradually dissolves it without the use of heat, and the process requires no attention after it has once been started. The process may be carried out as is described in (a), (b), or (c) below.

(a) The most simple method of applying this principle is to lay the weighed solid upon a piece of dry well-washed muslin. This is then tied up into a little bag, and is suspended in the upper part of the water contained in a jar of common stoneware or earthenware or of thick green glass. A glass rod placed across the mouth of the jar serves to hang the bag upon. The process of solution by this method is very rapid.

(b) A glass cylinder A (fig. 97) which is open at both ends (such as a broken beaker, flask, or bell-jar) has one end covered with muslin, which is fastened on by an india-rubber ring and is shown by the lower dotted line. This cylinder with a muslin bottom is supported in the jar by means of two pieces of glass rod or tube, bent as is shown at *c c c*.

Distilled water is poured into the jar until it buries the muslin to the depth of about half an inch. The weighed substance is then placed in the cylinder, and the whole is allowed to stand until the solid has been dissolved. With large quantities of substance this will usually require several hours; with smaller quantities about twenty minutes or half an hour will be necessary.

FIG. 97.



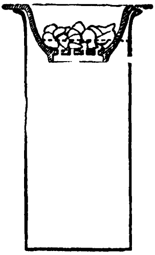
SOLUTION OF SOLID.

(c) A convenient substitute for the glass cylinder with muslin bottom is an earthenware colander (fig. 98). If the jar is too large a stoppered bottle of suitable size may be placed in it so as to raise the water level sufficiently to reach the substance.

The processes of solution and filtration may be performed simultaneously by laying a piece of filter-paper on the bottom of the colander, or upon the muslin before it is fastened on the cylinder. The presence of even the coarsest filter-paper will, however, considerably retard the process of solution.

Large quantities of substance are most conveniently dissolved by starting the process over night; the solution will then be completed by the morning. In this case the retardation caused by the filter-paper will be immaterial. The paper is, however, usually unnecessary; since any fine particles which have passed into the water will settle during the night, and the liquid can therefore be easily decanted from the sediment.

FIG. 98.



SOLUTION OF SOLID.

It must be borne in mind that the volume of the water is increased by the solution in it of a salt, and especially by salts which contain water of crystallisation. Hence the quantity of water which is placed in the jar must be less than the volume of solution required, and the liquid must be made up to the required volume after the solution has been effected.

1146. Preparation of Saturated Solutions of Solids.—A solution is said to be saturated, when it contains the largest amount of the substance which the liquid can dissolve at the temperature of the solution. The quantity which can be dissolved usually varies widely with the temperature, and in the case of solid substances commonly increases with rise of temperature. Saturation is always understood to be effected at ordinary atmospheric temperature in the following text.

The methods described in paragraph 1145 are very convenient for the preparation of saturated solutions. The solution is known to be saturated when the solid supplied to the upper part of the liquid ceases to be any further dissolved.

In the case of a finely-powdered substance, which will remain long suspended in water, a saturated solution may be easily obtained by shaking the powder up well with water. The powder is then allowed to subside, and the clear solution is decanted. The following solutions may be prepared in this way.

1. *Calcium sulphate Solution*, No. 16 (**1160**), is made by filling a Winchester-quart up to its shoulder with distilled water, then pouring in some plaster of Paris powder, stoppering the bottle, and shaking it well. The liquid will remain milky for a long time; and since the water is thus kept in contact with a large surface of the solid it will become saturated with $CaSO_4$ before the powder has settled.

As soon as the liquid has become perfectly clear by settling, the solution is decanted into another bottle for use. The sediment remaining in the first bottle is again shaken up with a fresh quantity of water and the bottle is set aside. It will then furnish a fresh supply of the clear solution when necessary.

2. *Lime-water*, No. 57 (**1162**), is prepared by introducing freshly-slaked lime into a Winchester-quart bottle, filled to its shoulder with *tap-water*, and proceeding precisely as is described for the preparation of calcium sulphate solution in the preceding paragraph.

SOLUTION OF GASES.

1147. Many gases, which are more or less soluble in water, are most conveniently applied as reagents in the dissolved condition. Those solutions which are most frequently used, such as solution of ammonia, of hydrogen chloride and of sulphur dioxide, can be readily and cheaply purchased.

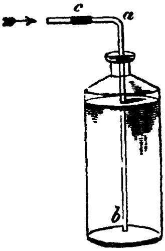
Of the remaining gaseous solutions, hydrogen sulphide solution should be kept in readiness, but the gas may be passed through the test liquid instead (**1127**); nitrogen tetroxide solution is replaceable by a freshly acidified solution of potassium or sodium nitrite; and as a substitute for chlorine-water, either bromine-water, or freshly acidified solution of bleaching-powder may be employed.

It will be seen, therefore, that the preparation of gaseous solutions is by no means indispensable.

1148. Preparation of Saturated Solutions of Gases.—All the solutions of gases (1149–1153) should be prepared in the open air or under a good draught, since the gases have an unpleasant smell and are injurious when inhaled.

Each gas is made to pass from the generating apparatus down a delivery-tube (*a b*, fig. 99) *to the bottom* of some cold distilled water, contained in a bottle which can be closed by a well-fitting stopper. The water is kept cool, if necessary, by immersing the bottle in a pan of cold water. Bubbles of gas are thus made to pass in a rapid stream through the whole height of the liquid, and a portion at least of each bubble is dissolved.

FIG. 99.



SATURATION OF WATER WITH GAS.

The solution is saturated as soon as the gas is not further dissolved. This may be judged in some cases by observing whether the bubbles cease to diminish in size during their ascent through the liquid. But a better test of saturation consists in withdrawing the delivery-tube, at once closing the bottle with the stopper or the thumb, and shaking it well. If pressure is felt from within, due to the liberation of some of the dissolved gas by the agitation, the solution is saturated. But if suction from within is felt, due to the gas filling the upper part of the bottle being dissolved, the liquid is not yet saturated with the gas.

1149. Saturated solution of Hydrogen sulphide (No. 36, 1161) is made by passing the gas from the apparatus described in paragraph 1125 through water (fig. 99) until the liquid is saturated (1148). The solution should be prepared in small quantity only, since the gas gradually undergoes decomposition, attended with deposition of sulphur, by oxygen dissolved from the air.

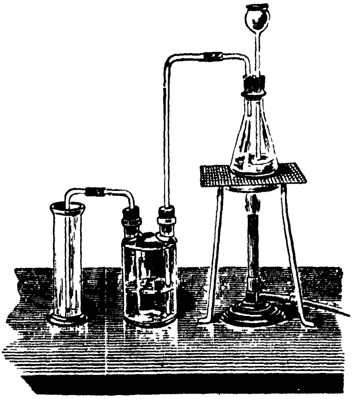
1150. Sulphurous acid Solution (No. 49, **1161**) is prepared as is described above (**1148**) by saturating water with sulphur dioxide gas. The gas may be made in the apparatus shown in figure 100 by the process described in paragraph **63**.

This solution is kept in stock for pharmaceutical purposes, and may therefore be readily purchased. Liquid SO_2 may also be purchased, and the gas which it evolves by spontaneous evaporation, when the valve is opened, may be employed for preparing the saturated solution.

1151. Chlorine-water (No. 60, **1162**) is made by passing chlorine into cold water until the liquid is bright yellow in colour. The apparatus for preparing the gas is shown in figure 100, and the process of preparation is described in paragraph **59**. The gas may also be obtained from a cylinder of liquid chlorine.

Chlorine-water is rapidly changed in daylight into solution of hydrochloric acid, with escape of oxygen. The liquid must therefore be kept in a perfectly dark closet, or in a bottle which is shielded from light by an opaque covering.

FIG. 100.



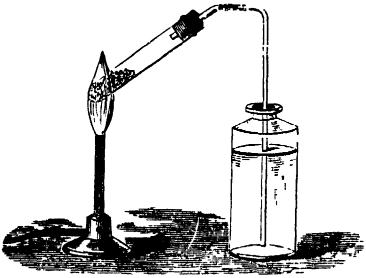
PREPARATION OF CHLORINE WATER.

For many purposes bromine-water (No. 43, **1161**) may be substituted for chlorine-water, and is much more easily prepared and preserved. Another substitute for chlorine-water is a little bleaching-powder shaken up with water in a test-tube, and acidified with dilute HCl.

1152. Nitrogen tetroxide Solution (No. 71, **1162**).—Lead nitrate in dry powder is heated in a test-tube fitted with a cork and delivery-tube (fig. 101). The reddish-brown gas which escapes is passed into dilute H_2SO_4.

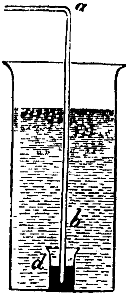
Care must be taken to maintain the heat uniformly after the fumes begin to be evolved, else the liquid may be sucked back into the hot tube by the contraction of the gas on cooling. For the same reason the delivery-tube must be removed from the liquid as soon as the heating is stopped. This solution need not be saturated with the gas.

FIG. 101.



SOLUTION OF NITROGEN TETROXIDE.

FIG. 102.



PASSING SILICON FLUORIDE INTO WATER.

1153. Hydrofluosilicic acid (No. 35, **1161**) is made by passing silicon fluoride gas into water. The gas is prepared in a flask fitted as is shown in figure 100. An intimate mixture of 50 grammes of dry, pure, finely-powdered fluorspar with 50 grammes of fine white sand is poured into this flask; 300 grammes of strong H_2SO_4 are then introduced through the funnel tube, and the acid is mixed with the powder by shaking it round in the flask.

SiF_4 is evolved when the flask is heated by a small flame from a rose-burner. The gas passes first through the two-necked bottle (fig. 100), which must be empty and dry inside. Thence it escapes through a bent tube *ab* (fig. 102), which is fitted upon the outlet tube of the bottle by an india-rubber joint. The end of the tube (*ab*) dips into mercury contained in a small beaker *d* (fig. 102), which stands in a large beaker containing 400 c.c. of water.

As soon as the silicon fluoride gas escapes from the mercury, and comes into contact with the water, it forms hydrofluosilicic acid which dissolves in the water, and silicic acid which remains suspended in the water as a gelatinous mass. The silicic acid would soon stop the escape of the gas, if the end of the delivery-tube were not protected from contact with water by being immersed in mercury.

When the current of gas slackens, the heat is raised until white fumes of H_2SO_4 begin to appear in the preparation flask. The process is then arrested, and the gelatinous matter is separated from the liquid in the beaker. This is effected by squeezing the liquid through fine muslin, and then filtering, if necessary, to make it quite clear. The silicic acid may be dried, heated strongly in a porcelain dish, and put by in a stoppered bottle for use as a reagent (No. 86, **1162**).

STORE-JARS AND BOTTLES.

1154. Stock of Solid Chemicals.—The broad-necked common green-glass jars with flat stoppers, which are used for preserves and confectionery, may be used as store-jars for large quantities of solid chemicals. These jars are very cheap and strong, and present the advantage over stoneware-jars that the quantity of material which they contain is seen at a glance. The flanged stoppers exclude dust and can be made air-tight by fastening a cork ring around their lower part. The complete exclusion of air is, however, seldom necessary. These jars can be purchased in several convenient sizes. Solids are readily removed from them by means of wooden spoons or horn scoops.

1155. Solids for Analysis.—The little white glass honey-jars with glass caps serve well for keeping smaller quantities of solids, and can be made air-tight by means of a cork strip fastened round the neck. These jars serve for storing the solid powders which are required for analysis in the laboratory (1169–1172): their necks are of the necessary width for the insertion of an ordinary watch-glass to remove substance.

1156. Reagents and Test-solutions.—Well-made German white-glass bottles can be purchased for the Reagents and Test-solutions. These bottles have flat-headed stoppers which protect the lip from dust, and each stopper has been ground into the bottle so as to ensure its fitting. Stopper and bottle are then prevented from permanently parting company by having an identical number etched upon them.

Accurately fitting glass stoppers should not be used in bottles containing solution of KHO or NaHO, as they are certain to be set fast. The use of a loosely fitting glass stopper, made to fit by surrounding it with a piece of rubber tube, obviates this difficulty.

The liquid Bench-reagents (1160) may be kept in narrow-necked bottles of 6 or 8 ounces capacity, and the solids in 4-ounce broad-necked bottles. The General reagents (1161, 1162) should be in 12-ounce bottles. The Test-solutions (1163, 1164) require larger bottles of about 32-ounce capacity: these solutions may be kept in stock in Winchester-quart bottles.

1157. Stock Bench-reagent Solutions.—The Bench reagent solutions (1160) may be conveniently kept in stock in an accessible position in the laboratory in large white-glass jars with glass taps below. These taps are ground into a tubulure near the bottom of the bottle. After the perfectly dry tap has been greased and inserted, it is fastened in the tubulure by applying externally a thick coating of paraffin wax. The plug and seat of the stopper require to be occasionally perfectly dried and then greased. This arrangement of the stock enables each student to fill his own bench-bottles when necessary.

LABELLING BOTTLES.

1158. Every Bottle in use in the Laboratory should be Labelled. Beginners are much assisted if the label bears not only the name but also the chemical formula of the substance which is contained in the bottle. It is also convenient to legibly number each bottle in the series. This number serves as a short means of reference. It also helps to keep the bottle in its place, so as to ensure its being easily found, if the edges of the shelves are numbered to correspond with the bottles which should stand on them.

The gummed labels should be stored in a dry place. When the label is used, the whole of the gummed surface should be wetted and made to adhere to the bottle by pressure with a clean cloth. The label is then allowed to dry thoroughly, and is protected by being brushed rapidly across with an ordinary broad flat and stiff gum-brush, which has just been taken out of a dish of melted paraffin-wax. A thin coating of the wax is thus made to entirely cover the label and to project for a short distance beyond its edge upon the glass surface.

A little practice will render it possible to lay on an even layer of the wax, which does not penetrate and stain the paper. If the paraffin is heated only slightly above its melting-point it will set too rapidly to penetrate and stain the paper.

This paraffin varnish, if properly applied, perfectly resists the action of water, acids and alkalis. In fact, it can only be removed or injured by mechanical means, by heat, or by such solvents as ether and turpentine.

Bottles containing the strong mineral acids commonly have the name or formula etched upon their surface. This is costly and it is unnecessary, since the paraffined paper label resists the action even of strong acids.

Names and formulæ painted on bottles with Brunswick black are also permanent in the laboratory.

LISTS OF REAGENTS AND CHEMICALS.

1159. In the Following Lists (1160–1165) each Reagent and Test-substance, which is required in the preceding Analytical Course, will be found.

In the First Column of the Table is a number which may be used for reference, and which serves to keep the bottle in its place on the shelf.

In the Second Column stands the name of the substance, and in the Third its chemical formula.

The subsequent columns give the strength of the solution, and the details of its preparation are added if the general methods described in paragraphs 1140–1146 do not apply.

An "s" following the reference numbers in paragraphs 1163, 1164 means that the substance is required in the solid state as well as in solution. A 4-ounce bottle containing some of the powdered solid should be placed near the bottle which contains the solution.

The directions for preparing liquid reagents have been already given in paragraphs 1140–1153. It is only necessary to add, that in stating the proportions of solid or liquid to water, required for the preparation of the solutions, the water always stands last; and that (*m*) signifies proportion *by measure* of liquids to water, and (*w*) proportion *by weight* of solids to water.

1160.—REAGENTS REQUIRED

1. Reference number.	2. Name. [The numbers in brackets refer to remarks correspondingly numbered in the sixth column.]	3. Formula.
Liquids.		
1.	Sulphuric acid (1),	H_2SO_4
2.	Hydrochloric acid (2),	HCl
3.	Nitric acid (3),	HNO_3
4.	Acetic acid (4),	$H\bar{A}$, or $HC_2H_3O_2$
5.	Ammonium chloride (5),	NH_4Cl
6.	Ammonia, or Ammonium hydrate (6),	NH_4HO
7.	Ammonium sulphide (7),	$(NH_4)_2S$
8.	Ammonium carbonate (8),	$(NH_4)_2CO_3$
9.	Ammonium oxalate,	$(NH_4)_2C_2O_4.H_2O$
10.	Potash, or Potassium hydrate,	KHO
11.	Potassium chromate,	K_2CrO_4
12.	Potassium ferrocyanide,	$K_4FeCy_6.3H_2O$
13.	* Potassium ferricyanide (9),	K_3FeCy_6
14.	† Sodium phosphate (10), or Hydric disodic phosphate, or Hydrogen disodium phosphate,	$Na_2HPO_4\ 12H_2O$
15.	Sodium carbonate,	$Na_2CO_3\ 10H_2O$ (crystals)
16.	Calcium sulphate,	$CaSO_4$
17.	Barium chloride,	$BaCl_2.2H_2O$
Solids.		
18.	Sodium bicarbonate (11),	$NaHCO_3$
19.	Borax (12),	$Na_2B_4O_7$
20.	Potassium chlorate,	$KClO_3$
21.	Test papers,	
22.	Reduction mixture (111)	Na_2CO_3+KCN

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* This reagent undergoes decomposition by exposure to light, and must not be kept

† Hydrogen sodium ammonium phosphate $(NaAmHPO)_4$ solution serves better for given above for Na_2HPO_4.

FOR EACH BENCH.

4. Weight of solid in grams to be dissolved in one "Winchester" of water.	5. Proportion of solid by weight (w.) and liquid by measure (m.) to water.	6. Remarks. [The small numbers refer to corresponding numbers in the second column.]
. . . .	1 : 5 m.	1. Pure strong H_2SO_4 must be poured in the proper proportion into water contained in a large thin beaker or an earthen jar, constantly stirring the water all the time: the right proportions are obtained by measurement of the height of the vessel (1140). The hot liquid is cooled by immersing the vessel in cold water, and is then poured into the store bottle. If the acid is pure, no white precipitate ($PbSO_4$) forms during dilution. 2. Must be colourless, and give after dilution no precipitate with either $BaCl_2$ or H_2S, and no colour with KCyS. 3. Must give no precipitate after dilution, with $BaCl_2$ or $AgNO_3$, added to separate portions, and no colour with KCyS. 4. Must give no precipitate with $BaCl_2$. 5. Must give no precipitate or coloration with Am_2S. 6. Must give only a very slight precipitate with lime water, no coloration with Am_2S, and separate portions acidified with HNO_3 must give no precipitates with $BaCl_2$ and $AgNO_3$. "Liquor ammoniæ fortissima," of 880° specific gravity, is bought. 7. Must be yellow, and give with acids H_2S and a white precipitate of S it must give no precipitate with solutions of Ca- or Mg-salts. 8. The solid Am_2CO_3 is dissolved by the second method (1145) in cold water, but in diluting, one-fourth of the "Winchester" must be filled with strong AmHO. 9. Must give no blue precipitate with Fe_2Cl_6 solution. 10. Must yield no precipitate with AmHO. 11. Dry finely-powdered "bicarbonate of soda." It must, after having been dissolved in excess of HNO_3, give no precipitate with $BaCl_2$ or $AgNO_3$, or $AmHMoO_4$ (578) and if evaporated with excess of HCl must leave no residue insoluble in dilute HCl (599) 12. The borax is best dried by heating it in a platinum or porcelain dish until after melting it has again become solid; this solid mass is then finely powdered in a mortar and kept in a stoppered bottle.
... ...	1 : 3 „	
. . . .	1 : 3 „	
... ...	2 : 1 „	
. 300 . .	1 : 8 w.	
... ...	1 : 8 m.	
. . . .	1 : 1 „	
... 400	1 : 5 w.	
. 100 . .	1 : 24 „	
... 300	1 : 8 „	
. 200 . .	1 : 12 „	
... 200	1 : 12 „	
. 200 . .	1 . 12 „	
... 200	1 : 12 „	
. 600 . .	1 : 4 „	
Saturated solution (1146)		
. 200 . .	1 12 „	
In small crystals.		
Blue and red litmus, and turmeric papers, in small strips.		

near a window. Dissolve a fragment of the solid (No. 93) each time it is required.
precipitation of Mg than does Na_2HPO_4 solution: it is prepared of the strength

1161.—REAGENTS FOR GENERAL USE,

Except in large laboratories, it will be unnecessary to keep these in the laboratory, may be made up to the right strength, as soon as they are

1. Reference number.	2. Name. [The small numbers in brackets refer to remarks correspondingly numbered in the fifth column]	3. Formula.
25.	Sulphuric acid (17)	H_2SO_4
26.	Hydrochloric acid (2)*	HCl
27.	Nitric acid (3)*	HNO_3
28.	† Platinum chloride	$PtCl_4$
29.	Alcohol, Rectified spirit (18)	C_2H_6O
30.	Tartaric acid (19)	$H_2\bar{T}$ or $H_2C_4H_4O_6$
31.	Acid sodium tartrate, Hydric sodic tartrate (20)	$NaH\bar{T}.H_2O$ $NaHC_4H_4O_6.H_2O$
32.	Slaked lime (21)	$Ca(HO)_2$
33.	Acetic acid (4)*	$H\bar{A}$ or $HC_2H_3O_2$
34.	Cobalt nitrate (22)	$Co(NO_3)_2\,6H_2O$
35.	Hydrofluosilicic acid (23)	H_2SiF_6
36.	Sulphuretted hydrogen solution Hydrogen sulphide (24)	H_2S-solution
37.	Oxalic acid	$H_2\bar{O}.2H_2O$ $H_2C_2O_4.2H_2O$
38.	Potassium sulphocyanide	$KCyS$
39.	Potassium nitrate	KNO_3
40.	Lead acetate (25)	$Pb\bar{A}_2.3H_2O$ $Pb(C_2H_3O_2)_2.3H_2O$
41.	Sodium acetate	$Na\bar{A}.3H_2O$ $NaC_2H_3O_2.3H_2O$
42.	Potassium cyanide (26)	KCy, or KCN
43.	Bromine-water (27) ‡	Br-water
44.	Stannous chloride (28)	$SnCl_2.2H_2O$
45.	Copper (29)	Cu
46.	Zinc (30)	Zn
47.	Steel (31)	Fe
48.	Silver nitrate	$AgNO_3$
49.	Sulphurous acid	H_2SO_3
50.	Copper sulphate	$CuSO_4.5H_2O$
51.	Magnesium sulphate	$MgSO_4.7H_2O$
52.	Mercuric chloride	$HgCl_2$
53.	Gold chloride	$AuCl_3$
54.	Ammonium acetate	$Am\bar{A}$

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* See last column on the preceding page.

† Must be used in very small quantity only, being an expensive reagent.

‡ Sodium hypochlorite (Na_2Cl_2O), the "Liquor Sodæ Chlorinatæ" of commerce may be used, but does not keep well.

FOR THE DETECTION OF METALS.

reagents in store in quantity. The 12 oz. bottles in which they are contained empty, by the proportions stated below in column 4.

4. Proportion by weight of solid to water: and weight in grams for an 18 oz bottle in square brackets.
. . Strong pure.
... " "
. . " "
... 1 : 30 [15 grams]
. . Strong.
... 1 : 10 [45 grams]
... Solid.
. . Strong.
... 1 : 12 [40 grams].
. . See (1153).
.. See (1149).
. . 1 : 12 [40 grams].
... 1 · 100 [5 grams].
. . Solid.
... 1 : 12 [40 grams].
. . Solid.
.. Solid.
... 1 : 12 [40 grams].
... 1 : 25 [20 grams]
. . See 1026).
... 1 : 12 [40 grams].
. . 1 : 12 [40 grams].
... 1 : 20 [30 grams].
. . 1 : 30 [15 grams].
... 1 : 12 [40 grams]

5\.

Remarks.

[The numbers in this column refer to those in brackets in the second column.]

17. Must be colourless and form no brown ring when poured below some $FeSO_4$ solution (544).
18. Strong rectified methylated spirit, which must leave no residue on evaporation, and remain clear on dilution.
19. The solution should be mixed immediately after preparation with several drops of carbolic acid to prevent a vegetable growth forming in it: or better still some crystallised $H_2\overline{T}$ should be dissolved when wanted.
20. Dissolve 10 grams of $H_2\overline{T}$ in 100 c c * of water, divide this solution into equal parts, exactly neutralise (103) one part by heating it nearly to boiling and stirring in solid Na_2CO_3 in powder; add to this the other part, cool and dilute to 150 c c. A few drops of carbolic acid should be added to the solution.
21. Pieces of freshly-burnt lime are placed on a plate, and water is poured upon them until they begin to appear moist on their surface; the superfluous water is then drained off, and as soon as the lime has crumbled to powder, it is placed in a broad-mouthed stoppered bottle.
22. The bottle should be closed with a loosely-fitting india-rubber stopper, perforated, and with a glass tube passed through it and dipping into the solution; when a drop of the solution is required, the upper end of the glass tube is closed by the finger, and a drop is delivered from the opposite end by slightly relaxing the pressure of the finger.
23. The solution must give no precipitate with $Sr(NO_3)_2$ solution.
24. This solution must not darken on adding AmHO.
25. A little $H\overline{A}$ must be added to this solution to make it clear.
26. Solution of KCy is made (1 : 12); it decomposes so readily that the solution is best made immediately before use by heating a small piece of solid KCy with distilled water.
27. A few drops of Br dissolved by well shaking with water
28. Crystals of $SnCl_2$ should be dissolved by heating them with water containing some HCl; the solution should be kept in a well-stoppered bottle containing pieces of granulated or block tin.
29. In strips cut from thin copper-sheet.
30. In strips or rods, or granulated; it must be proved to be free from As (315, 316, or 317).
31. Common knitting-needles broken into short lengths, and kept in a bottle containing pieces of quicklime to prevent rusting.

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* c.c. is the contraction for cubic centimetre. See weights and measures (1175).

1162.—REAGENTS FOR GENERAL USE,

Refer to the statement under

1. Reference number.	2. Name [The small numbers in brackets refer to corresponding numbers in the fifth column]	3. Formula.
57	Lime water	$Ca(HO)_2$
58.	Lead acetate in potash (32)	$Pb\bar{A}_2+KHO$
59	Potassium dichromate	$K_2Cr_2O_7$.
60	Chlorine water	Cl-solution
61	Ferric chloride (33)	Fe_2Cl_6
62	Ferrous sulphate (34)	$FeSO_4.7H_2O$
63	Potassium iodide	KI
64	Starch (35)	
65.	Indigo-solution (36)	
66.	Manganese dioxide (37)	MnO_2
67	Potassium dichromate (38)	$K_2Cr_2O_7$.
68.	Ether (methylated)	$(C_2H_5)_2O$
69	Carbon disulphide	CS_2
70	Potassium nitrite (39)	KNO_2
71	Nitrogen tetroxide solution	N_2O_4-solution
72	Ammonium molybdate (40)	$(NH_4)HMoO_4$
73	Calcium fluoride	CaF_2
74	Potassium disulphate	$KHSO_4$
75	Microcosmic salt, or Hydric ammonic sodic phosphate (40 a)	$NaAmHPO_4\ 4H_2O$
76.	Wax, or paraffin	
77.	Calcium chloride	$CaCl_2\ 6H_2O$ (crystals)
78.	Potassium chloride	KCl
79.	Marble	$CaCO_3$
80*.	Distilled water (41)	H_2O
81.	Pure sodium hydrate (42), pure soda	NaHO
82.	Fusion mixture (43)	$Na_2CO_3+K_2CO_3$
83.	Barium carbonate (44)	$BaCO_3$
84.	Solution of sodium acetate in dilute acetic acid (45)	$Na\bar{A}+H\bar{A}$
85.	Sodium nitrate (46), solid,	$NaNO_3$
86.	Silica, or finely powdered white sand (1153)	SiO_2
87.	Hydrofluoric acid (47)	HF
88.	Zinc sulphate (See No 111)	$ZnSO_4.7H_2O$
89.	Calcium carbonate, pure (970)	$CaCO_3$
90.	Ammonium chloride, pure, in powder (1031)	NH_4Cl
91.	Barium peroxide, in fine powder	BaO_2
92.	Alcohol, sp gr. 0 83	C_2H_6O
93.	Potassium ferricyanide, solid, in small pieces (1160 13)	K_3FeCy_6
94.	Lead nitrate, in small pieces	$Pb(NO_3)_2$

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* Nos. 80-94 are certain special

FOR THE DETECTION OF ACID-RADICLES.

the heading of paragraph 1161.

4 Proportion by weight of solid to water. and weight in grams for a 18 oz. bottle in square brackets.
. See (1146)
. 1 : 24 [20 grams].
... See (1151).
. 1 . 24 [20 grams].
... Solid
. 1 : 60 [8 grams].
... Solid.
... Solid.
. Solid.
... 1 · 24 [20 grams].
. See (1152).
. In powder
... In small pieces.
. In small crystals
... Solid in pieces.
. 1 : 12 [40 grams].
... 1 : 12 [40 grams].
. In pieces as large as a pea

5.

Remarks.

[The numbers in this column refer to corresponding numbers in the second column].

32. To some of the $Pb\bar{A}_2$ solution (No 40) KHO solution is added until, on warming, the precipitate at first formed is just redissolved paper dipped into this liquid is a very delicate test for H_2S (512)
33. The solution should not contain any free acid: to remove acid AmHO is added until the further addition of *a single drop* gives a reddish-brown precipitate of $Fe_2H_6O_6$ in the solution.
34 $FeSO_4$ solution rapidly oxidises in the air, hence the $FeSO_4$ should be kept in the solid state as small green crystals, which should not show a yellow coating in any part, these are dissolved when required by crushing and shaking with cold water
35. Starch solution rapidly changes, it is best therefore to keep the starch as a powder Starch solution is made by stirring 2 grams of this powder, which has been made into a paste with 10 c c of cold water, into 100 c c. of boiling water and cooling.
36 Made by dissolving indigo-carmine in water.
37 Should be kept in fine powder, it must not evolve Cl or CO_2 when warmed with strong H_2SO_4.
38 In small pieces, or in powder
39. The solution is prone to change, and should be made in small quantity only it must evolve copious red fumes when mixed with H_2SO_4
40. Make the solution with the following proportions:—1 gram of Am_2MoO_4 is dissolved in 12 5 c c. of strong AmHO which has been previously mixed with an equal quantity of water, the solution is allowed to stand (if necessary) till clear, then poured off into 50 c.c of strong HNO_3: the liquid will become hot, and should be allowed to stand until it is cool before being used.

40 *a*. The HNO_3 solution of this salt must yield no precipitate with $AgNO_3$
41. Must leave no residue on evaporation, in separate portions no precipitates must be caused by $BaCl_2$, $AgNO_3$, or $Am_2C_2O_4$, neither must any precipitate or even dark coloration be produced by addition of Am_2S
42. The proportion by weight should be 1 10 of water, it is best kept in green glass bottles, as it slowly dissolves Pb from white flint glass. The solution must not become dark in colour when mixed with H_2S, nor give a gelatinous precipitate $Al_2(HO)_6$ when mixed with excess of AmCl solution.
43. Dry finely powdered Na_2CO_3 and K_2CO_3 are intimately mixed in the proportion of 53 69 by weight, and kept in a stoppered bottle
44. Pure powdered $BaCO_3$ is either purchased, or is made by precipitating $BaCl_2$ solution completely with Am_2CO_3 or Na_2CO_3 solution, and washing the precipitate well by decantation. This powder is then mixed with distilled water to the consistency of thin cream.
45. Dissolve 20 grams of $Na\bar{A}$ in 60 c c. of distilled water, and add to the solution 40 c.c. of strong $H\bar{A}$.
46. Made by neutralising strong hot Na_2CO_3 solution with HNO_3 and evaporating to dryness Sold also as Chili saltpetre.
47. Purchased and kept in gutta-percha bottles.

reagents entered apart.

1163.—SOLUTIONS FOR THE

Each bottle should bear on its label the number, name, and formula of
solid of each of those substances whose number has a small (s) affixed to
bottles should also carry on their label the name, number, and formula of the

1. Reference number	2 Name. [The numbers in brackets refer to similarly numbered remarks in the last column.]	3. Formula.
100.s	Potassium chloride	KCl
101 s	Ammonium chloride	$AmCl$
102 s.	Sodium chloride.	$NaCl$
103.s.	Magnesium sulphate	$MgSO_4\ 7H_2O$
104.	Barium chloride	$BaCl_2.2H_2O$
105.	Strontium nitrate	$Sr(NO_3)_2.4H_2O$
106.	Calcium chloride	$CaCl_2.6H_2O$
107 s.	Alum, or Aluminium sulphate	$Al_2(SO_4)_3$
108.	Ferric chloride (*a*)*	Fe_2Cl_6
109.s.	Ferrous sulphate (48)	$FeSO_4\ 7H_2O$
110.s.	Chrome alum, or Chromium potassium sulphate	$CrK(SO_4)_2.12H_2O$
111 s	Zinc sulphate	$ZnSO_4.7H_2O$
112.s.	Manganese chloride (*a*)	$MnCl_2$
113.s.	Nickel sulphate	$NiSO_4.7H_2O$
114.	Cobalt nitrate	$Co(NO_3)_2.6H_2O$
115.s.	Mercuric chloride	$HgCl_2$
116.s.	Lead acetate (49)	$Pb\bar{A}_2.3H_2O$
117.s.	Bismuth nitrate (50)	$Bi(NO_3)_3.5H_2O$
118.s.	Copper sulphate	$CuSO_4.6H_2O$
119.s.	Cadmium sulphate (*a*)	$CdSO_4.4H_2O$
120.s.	Arsenious oxide (*a*) (51) Solution in dilute HCl	As_2O_3
120.	Arsenious oxide (*a*) (52) Solution in water	
121.s.	Sodium arsenate	$Na_2HAsO_4.12H_2O$
122.s.	Antimonious chloride (53) †	$SbCl_3$
123.s.	Stannous chloride (54)	$SnCl_2.2H_2O$
124.	Stannic chloride (55)	$SnCl_4$
125.	Silver nitrate	$AgNO_3$
126.	Mercurous nitrate (56)	$Hg_2(NO_3)_2.H_2O$

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* *a* signifies that the solid substance is

† Solid Sb_2O_3 or $K(SbO)\overline{T}$ (Tartar emetic)

REACTIONS OF THE METALS.

the substance. A small 4 oz. wide-mouthed bottle, containing the powdered it, should be kept near the corresponding solution. These wide-mouthed substance.

4. Weight of solid in grams to be dissolved in one "Winchester."	5. Proportion by weight of solid to water.
100	1 : 25
200	1 : 12
50	1 : 50
50	1 : 50
25	1 : 100
300	1 : 8
200 (in crystals)	1 : 12
100	1 : 6
25	1 : 100
25	1 : 100
50	1 : 50
25	1 : 100
25	1 : 100
50	1 : 50
50	1 : 50
50	1 : 50
50	1 : 50
25	1 : 100
50	1 : 50
25	1 : 100
10	1 : 250
50	1 : 50
25	1 : 100
25	1 : 100
25	1 : 100
25	1 : 100

6

Remarks.

[These numbers correspond to the small reference numbers in brackets in the second column.]

48 The solution must be made acid with a little H_2SO_4, and some clean iron nails kept in it; or better, a small quantity is freshly prepared when required, see No. 62 (1162)

49. A little $H\bar{A}$ must be added to this solution to make it clear

50. 25 grams of $Bi(NO_3)_3$ must be dissolved by heating with 25 c c of strong HCl diluted with 25 of water; this solution is cooled and poured into the bottle, and the measure made up by pouring in dilute HCl containing one-twentieth of its volume of strong HCl

51 Dissolve 10 grams of As_2O_3 by heating it with 50 c.c. of strong HCl mixed with 50 c.c. of water, and dilute to the Winchester-quart

52 Boil excess of As_2O_3 for several minutes with 2000 cc. of water, and filter into the quart-bottle.‡

53 28 grains of crystallised $SbCl_3$ are dissolved in 25 c.c. of strong HCl mixed with 25 c c. of water, and diluted to the quart‡ with HCl mixed with four times its measure of water.

54 Boil 25 grams of $SnCl_2$ with 50 c c. of strong HCl mixed with 50 c c of water, as soon as it has dissolved to a clear solution dilute to the quart‡. Scraps of Sn must be kept in the bottle.

55. Heat 25 grams of $SnCl_2$ with 25 c.c. of strong HCl and 100 c c of water, whilst constantly stirring, add $KClO_3$ to the hot solution until the liquid turns yellow and Cl is smelt, boil off the Cl and dilute to the quart‡.

56 Dissolve by warming 25 grams of $Hg_2(NO_3)_2$ with 6 c.c. of strong HNO_3 diluted with 114 c.c. of water, then pour water into this solution until it is diluted to a quart‡. Keep Hg in the bottom of the bottle.

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not in the crystalline condition.

may be used for the blowpipe reaction.

‡ Winchester-quart.

1164. SOLUTIONS FOR THE

Refer to the note at the head of the preceding Table, which is also amongst the reagents (1160–1162) and the solutions for the reactions of

1. Reference Number.	2. Name. [The small numbers in brackets refer to the corresponding numbers in the sixth column.]	3. Formula.
130.	Sodium sulphate	$Na_2SO_4.10H_2O$
131 s.	Sodium carbonate (57)	$NaHCO_3$
	Marble (calcium carbonate) (57a)	$CaCO_3$
132. s.	Ferrous sulphide (58)	FeS
	Ammonium sulphide (58a)	Am_2S
133 s.	Acid sodium sulphite	$NaHSO_3$
134.	Sodium thiosulphate	$Na_2S_2O_3.5H_2O$
135.	Sodium hypochlorite (59)	Na_2Cl_2O
136 s.	Potassium nitrite (*a*)	KNO_2
137. s.	Potassium nitrate [Solid Lead nitrate]	KNO_3 $[Pb(NO_3)_2]$
138 s.	Potassium chlorate	$KClO_3$
139 s.	Sodium chloride (60)	$NaCl$
140 s.	Sodium bromide	$NaBr$
141 s.	Potassium iodide	KI
142.	Sodium phosphate	$Na_2HPO_4.12H_2O$
143	Sodium arsenate (61)	$Na_2HAsO_4.12H_2O$
144 s.	Borax (62)	$Na_2B_4O_7.10H_2O$
145	Potassium bichromate	$K_2Cr_2O_7$
146 s.	Sodium silicate (63)	Na_2SiO_3
	Silica (white sand)	SiO_2
147. s.	Fluor-spar (calcium fluoride)	CaF_2
148.	Hydrofluosilicic acid (64)	H_2SiF_6
149 s.	Potassium cyanide (65)	KCy
150.	Potassium ferrocyanide	$K_4FeCy_6.3H_2O$
151.	*Potassium ferricyanide	K_3FeCy_6
152.	Potassium sulphocyanide	$KCyS$
153 s.	Ammonium oxalate (66)	$Am_2C_2O_4.2H_2O$
154. s.	Tartaric acid (67)	$H_2\overline{T}$
	Sodium tartrate (68)	$Na_2\overline{T}.2H_2O$
155. s.	Sodium acetate (69)	$Na\bar{A}.3H_2O$

* See No. 13 (1160).

REACTIONS OF THE ACID-RADICLES.

applicable to this. Many of the solutions in this list are already prepared
metals (1163).

4. Weight of solid in grams to be dissolved in one Winchester-quart.	5. Proportion by weight of solid to water.	6. Remarks. [The numbers correspond to the small reference numbers in brackets in the second column.]
25	1 : 100	57. The $NaHCO_3$ is required only in the solid form, and can be taken from the reagent bottle on the working bench, No 18 (1160). 57a. The marble is kept in small pieces as large as a pea, it is found in reagent bottle No. 79 (1162)
... 100 ...	1 : 25	58 The FeS is kept as a solid in small pieces as large as a pea.
. 100 .	1 : 25	58a The Am_2S can be taken from reagent bottle No 7 (1160) on the working bench.
. 100 .	1 : 25	59. Strong "Liquor Sodæ Chlorinatæ," diluted with an equal measure of water.
... 100 ...	1 : 25	
. 50 .	1 : 50	60 No 102, paragraph (1163)
... 50 ...	1 : 50	61 No 121, paragraph (1163). 62 No 19, paragraph (1160).
50 .	1 : 50	63 The solution is made by dissolving 100 grams of the thick syrup, sold as "soluble glass," in water, and diluting to the Winchester-quart The solid substance to be used is sand finely ground.
... 12 ...	1 : 200	
. 50 .	1 . 50	
... 50 ...	1 : 50	64 No 35, paragraph (1161)
... 25 ...	1 : 100	65 No 42, paragraph (1161)* The solution decomposes by keeping, a little solid is dissolved when required.
In fine powder.		66 No 9, paragraph (1160) 67. No 30, paragraph (1161).
. 50 .	1 . 50	68. 123 grams of $H_2\bar{T}$ are dissolved by heat in 500 c c of water, the hot solution is exactly neutralised with solid Na_2CO_3, and then diluted to a Winchester-quart.
... 25 ...	1 100	
. 25 .	1 . 100	69. A little of the solid substance (No 41, 1161) is dissolved, when required, in water.
... 25 ...	1 . 100	
. 100 .	1 : 25	
... ...	1 : 10	

1165. SPECIAL TEST-SUBSTANCES AND REAGENTS FOR THE RARER ELEMENTS AND ORGANIC SUBSTANCES.

Only those substances are entered here which do not occur in the preceding lists.

I INORGANIC TEST-SUBSTANCES

Thallium sulphate, . . .	Tl_2SO_4
Sodium tungstate, .	Na_2WO_4
Palladium chloride, .	$PdCl_2$
Ammonium molybdate,	Am_2MoO_4
Iron selenide, ...	FeSe
Potassium selenate, . .	K_2SeO_4
Lead telluride, . . .	PbTe
Potassium tellurate, . .	K_2TeO_4
Uranium nitrate, ...	$UO_2(NO_3)_2$
Indium sulphate, ...	$In_2(SO_4)_3\ 9H_2O$
Beryllium sulphate, .	$Be_2(SO_4)_3$
Titanium oxide, . . .	TiO_2
Sodium vanadate, .	$NaVO_3$
Lithium chloride, . .	LiCl
Rubidium chloride, .	RbCl
Cæsium chloride, ...	CsCl
Sodium phosphite,	Na_2PHO_3
Sodium hypophosphite,	$NaPH_2O_2$
Potassium permanganate	$K_2Mn_2O_8$
Potassium manganate .	K_2MnO_4

REAGENTS.

Mercuric cyanide solution, ...	$HgCy_2$
Potassium bicarbonate, solution, ...	$KHCO_3$
Tin, granulated, ...	Sn
Hydrogen peroxide, dilute acid solution, . . .	H_2O_2
Calcium sulphate, in fine powder, ...	$CaSO_4$

II. ORGANIC TEST-SUBSTANCES.

Sodium formate, . . .	$NaHCO_2$
Ammonium succinate, .	$Am_2C_4H_4O_4$
Ammonium benzoate, .	$AmC_7H_5O_2$
Malic acid, ...	$C_4H_6O_5$
Sodium citrate, ...	$Na_3C_6H_5O_7.2H_2O$
Salicylic acid, ...	$C_7H_6O_3$
Tannic acid, ...	$C_{14}H_{10}O_9$
Gallic acid, ...	$C_7H_6O_5$
Meconic acid, ...	$C_7H_4O_7.3H_2O$
Ammonium urate, . . .	$AmC_5H_3N_4O_3$
Hippuric acid, ...	$C_9H_9NO_3$
Picric acid, ...	$C_6H_3(NO_2)_3O$
Quinine sulphate, ...	$C_{20}H_{24}N_2O_2.H_2SO_4$
Cinchonine sulphate, . .	$C_{19}H_{22}N_2O.H_2SO_4$

II ORGANIC TEST-SUBSTANCES—*Contd.*

Morphine hydrochlorate, .	$C_{17}H_{19}NO_3.HCl$
Strychnine, ...	$C_{21}H_{22}N_2O_2$
Narcotine, . . .	$C_{22}H_{23}NO_7$
Brucine, ...	$C_{23}H_{26}N_2O_4$
Caffeine, . . .	$C_8H_{10}N_4O_2\ H_2O$
Turpentine, ...	$C_{10}H_{16}$
Benzene, ...	C_6H_6
Chloroform, ...	$CHCl_3$
Iodoform, ...	CHI_3
Methyl alcohol, ...	CH_4O
Ethyl alcohol, ...	C_2H_6O
Glycerol, ...	$C_3H_8O_3$
Phenol, ...	C_6H_6O
Hydroquinone . .	$C_6H_6O_2$
Pyrogallol, ...	$C_6H_9O_3$
Aldehyde, ...	C_2H_4O
Chloral hydrate, ...	$C_2HCl_3O.H_2O$
Benzaldehyde, ...	C_7H_6O
Acetone, ...	C_3H_6O
Urea, ...	CH_4N_2O
Aniline, ...	C_6H_7N
Ether, ...	$C_4H_{10}O$
Ethyl acetate, ...	C_2H_5A
Carbon disulphide, ...	CS_2
Nitrobenzene, ...	$C_6H_5(NO_2)$
Glucose (Dextrose), ...	$C_6H_{12}O_6$
Levulose, ...	$C_6H_{12}O_6$
Cane Sugar, ...	$C_{12}H_{22}O_{11}$
Maltose, ...	$C_{12}H_{22}O_{11}.H_2O$
Lactose, ...	$C_{12}H_{22}O_{11}.H_2O$
Cellulose, ...	$(C_6H_{10}O_5)n$
Starch powder, ...	$(C_6H_{10}O_5)n$
Dextrin, ...	$(C_6H_{10}O_5)n$
Albumen, ...	White of egg
Gelatin, ...	Isinglass

REAGENTS.

Mercuric nitrate, neutral solution, ...	$Hg(NO_3)_2$
Quicklime, in powder, . .	CaO
Potassium bicarbonate, solution, ...	$KHCO_3$
Iodic acid, solution (1 : 15),	HIO_3
Iodine, in small fragments,	—
Yeast from a brewer, . .	—
Rochelle salt, ...	$KNaT.4H_2O$
Rosaniline, ...	—
Magenta, ...	—
Zinc chloride, ...	$ZnCl_2$
Ammonium iodide, . . .	NH_4I
Sodium nitroprusside, . .	—

1166. CHEMICALS REQUIRED FOR SECTION II.

In the following list those substances which are used only for Section II. are marked by letters of the alphabet. The bottles containing these substances should be arranged apart in alphabetical order. Against others a number is placed; this indicates that the substance is used for analytical purposes, and will therefore be found, together with any necessary descriptive remarks, in one of the preceding lists (1160–1164)

Reference.	Name.	Formula.	Remarks.
A.	Mercuric oxide . .	HgO	The orange-coloured *precipitated* oxide is best.
20.	Potassium chlorate . .	$KClO_3$	—
B	Manganese dioxide . .	MnO_2	Commercial oxide in powder.
C.	Wood charcoal . .	C	In pieces as large as a hazel-nut.
57.	Lime water	$Ca(HO)_2$	—
D.	Sulphur	S	Pieces of roll sulphur as large as a pea.
21	Litmus and turmeric papers .	—	—
59.	Potassium dichromate .	$K_2Cr_2O_7$	Solution
E	Phosphorus	P	Pieces as large as half a pea, kept in a stoppered bottle under water
F	Granulated zinc . . .	Zn	Not necessarily pure.
26	Hydrochloric acid . .	HCl	Strong, pure
G.	Calcium chloride . .	$CaCl_2$	Fused, solid, in small pieces
79	Marble	$CaCO_3$	In small pieces
I.	Hydrochloric acid . .	HCl	Strong, commercial.
J	Litmus solution .	—	—
K	Copper	Cu	Turnings, clippings, or filings
L	Nitric acid	HNO_3	Strong, commercial.
63	Potassium iodide .	KI	Solution.
64.	Starch powder .	—	—
132s	Ferrous sulphide . .	FeS	Small pieces.
116	Lead acetate . . .	PbA_2	See also 58.
M	Ammonium chloride .	NH_4Cl	Solid, commercial.
32.	Slaked lime . . .	$Ca(HO)_2$	—
N.	Strong ammonia solution . .	NH_4OH	Liquor ammoniæ fortissima.
O.	Oxalic acid . . .	$H_2C_2O_4.2H_2O$	Solid, in crystals.
P.	Sulphuric acid . .	H_2SO_4	Strong, commercial.
Q	Sodium hydrate . .	$NaHO$	Strong solution in water
R.	Manganese dioxide .	MnO_2	In small pieces
S.	Oil of turpentine . . .	—	—
T.	Dutch metal	—	Leaf from a small book.
U.	" Turkey red " . . .	—	In strips
V.	Sodium chloride . .	$NaCl$	In pieces as large as a hazel-nut broken from a lump of rock salt.
3	Nitric acid	HNO_3	
48	Silver nitrate . . .	$AgNO_3$	
W.	Ammonium nitrate . .	NH_4NO_3	Solid, in small pieces.
92	Alcohol . . .	C_2H_6O	Sp grav. 0·83.
43.	Bromine water . . .	Br	
X.	Soda lime	—	Pieces size of millet seed.
41.	Sodium acetate . . .	$Na\bar{A}\ 3H_2O$	Crystallised.
6.	Ammonium hydrate .	NH_4HO	Solution.
9.	Ammonium oxalate .	$(NH_4)_2C_2O_4$	"
17.	Barium chloride . . .	$BaCl_2$	"
7	Ammonium sulphide .	$(NH_4)_2S$	"
39.	Potassium nitrate . .	KNO_3	Solid.

1167. CHEMICALS REQUIRED FOR SECTION III.

With few exceptions,* these substances are required either for analytical purposes, and are tabulated in paragraphs **1160-1164**, where they may be found by the number placed in the first column; or they are already entered in the list of substances required for experiments on the gases (**1166**), where they may be found by the reference letter in the first column. A letter (s) affixed to the number of reference indicates that the substance is required in the solid condition. See note at heading of paragraph **1163**.

Number or letter of reference	Name	Formula.
89.	Potassium nitrate	KNO_3
118s.	Copper sulphate	$CuSO_4\ 5H_2O$.
79.	Marble	$CaCO_3$
2	Hydrochloric acid	HCl
45	Copper	Cu.
3	Nitric acid	HNO_3.
17.	Barium chloride	$BaCl_2.2H_2O$.
3	Ammonium carbonate	Am_2CO_3.
—	*Filter-paper	In sheet, or cut
1.	Sulphuric acid	H_2SO_4.
A.	Mercuric oxide	HgO.
M.	Ammonium chloride	NH_4Cl.
100s	Potassium chlorid	KCl
18.	Sodium carbonate	Na_2CO_3
—	*Barium sulphate	$BaSO_4$.
B.	Manganese dioxide	MnO_2
19	Borax	$Na_2B_4O_7$.
62s	*Ferrous sulphate	$FeSO_4\ 7H_2O$.
102	Sodium chloride	$NaCl$.
—	Wood charcoal	C.
116s	Lead acetate	$PbA_2\ 3H_2O$.
118s.	Copper sulphate	$CuSO_4\ 5H_2O$.
6	Ammonium hydrate	NH_4HO.
21	Litmus-paper	—
	Turmeric-paper	—
—	*Zinc oxide	ZnO.

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* The exceptions are filter-paper (1121), wood charcoal (1123), zinc oxide, and barium sulphate.

SUBSTANCES TO BE GIVEN FOR ANALYSIS BY THE PRECEDING TABLES.

It must be understood that the substances mentioned below are merely brought forward as examples of what may be given to the student for analysis. The teacher will use his own judgment in adopting and extending the list.

SUBSTANCES FOR ANALYSIS WHILE TRYING THROUGH THE REACTIONS FOR METALS AND ACID-RADICLES.

1168. After working through the reactions for a Group of Metals or Acid-radicles, a few unknown substances containing only *one* member of the Group are tested; the number of members present is gradually increased in the substances which are subsequently given. It is well to keep these substances in the dissolved state (**1163, 1164**), since they are intended mainly to afford practice in separation and detection according to the Group-tables; the time spent by the student in preparing the solution would therefore be wasted. Occasionally, however, a solid substance may be given, in which the metal or Acid-radicle present is to be detected by blowpipe tests, or by other tests made on the solid substance. The following will serve as examples :—

Group V.	*Group IV.*	*Group III.b.*
Solution of NaCl	Solution of $BaCl_2$ $2H_2O$	Solution of $ZnSO_4.7H_2O$
" " NH_4Cl	" " $Sr(NO_3)_2.4H_2O$	" " $MnCl_2$
" " KCl	" " $CaCl_2.6H_2O$	" " $NiSO_4.7H_2O$
" " NaCl+KCl	" " $Sr(NO_3)_2.4H_2O$ $+CaCl_2.6H_2O$	" " $Co(NO_3)_2.6H_2O$
" " $NaCl+NH_4Cl+$ KCl	" " $BaCl_2.2H_2O+$ $Sr(NO_3)_2.4H_2O$ $+CaCl_2.6H_2O$	" " $MnCl_2+ZnSO_4$ $7H_2O$
" " $NaCl+NH_4Cl$	" " $BaCl_2.2H_2O+$ $Sr(NO_3)_2.4H_2O$	" " $Co(NO_3)_2$ $6H_2O+$ $NiSO_4$ $7H_2O+$ $ZnSO_4.7H_2O$
" " $MgSO_4.7H_2O+$ NaCl+KCl		&c., &c., &c.
Solids: NaCl, NH_4Cl, KCl, AmCl+NaCl, &c.		*Solids*: $MnCl_2$, $NiSO_4.7H_2O$, $Co(NO_3)_2$ $6H_2O$. $ZnSO_4.7H_2O$, &c.

SUBSTANCES FOR ANALYSIS BY THE DIRECTIONS CONTAINED IN SECTION V.

1169. Any one of the solutions named in paragraphs **1163, 1164** may be given for analysis by Section V.: some of these are neutral, some acid, and others alkaline in reaction. The following more difficult solutions, with acid or alkaline reaction, may also be added to the list:—

Acid.	*Alkaline.*
$Ca_3(PO_4)_2$, "Bone-ash" dissolved in dilute HCl	$KSbO_3$ dissolved in water
BaC_2O_4 dissolved in dilute HCl.	Na_2SiO_3, solution of "soluble glass."
$Mg(BO_2)_2$* ,, ,, HCl.	Na_2SnO_3, "preparing salt" of the dyer.
$BaCrO_4$,, ,, HCl.	

Any of the solid salts which were dissolved in order to prepare the solutions in paragraphs **1163, 1164** may be given for analysis by paragraphs **932**, *et seq.*

As examples of substances possessing metallic appearance (**969**), the following may be mentioned as suitable for analysis:—

Zn, in pieces or filings.	NiAs, Kupfernickel.
Pb ,, ,,	Graphite, or Black lead.
FeS_2, Iron pyrites.	Iron filings.

As insoluble substances which require to be examined by paragraphs **970**, *et seq.*, the following may be given for analysis:—

$BaSO_4$.	CaF_2, Fluor spar.
$PbSO_4$.	$FeCr_2O_4$, Chrome Iron ore.
AgCl.	S, as pieces of roll sulphur.
SnO_2, Tinstone.	C, as powdered wood charcoal or plumbago.

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* Made by mixing hot solutions of $Na_2B_4O_7$ and $Mg(NO_3)$.

SOLID SUBSTANCES TO BE ANALYSED BY THE PRELIMINARY EXAMINATION IN SECTION VI.

1170. For the Preliminary Examination for Metals (989) :—

Simple			*Complex.*	
NH_4Cl $HgCl_2$ As_2O_3 KNO_3 Cr_2O_3	$Pb(NO_3)_2$ $NaCl$ $BaCl_2\ 2H_2O$ $Sr(NO_3)_2\ 4H_2O$ $CuSO_4\ 5H_2O$ (See *note*)	$ZnSO_4\ 7H_2O$ $MnCl_2$ $Co(NO_3)_2\ 6H_2O$ $NiSO_4\ 7H_2O$ SnO_2	$AmCl+NaCl$ $HgCl_2+BaCl_2\ 2H_2O$ $MnCl_2+KCl$ $NiSO_4.7H_2O+ZnSO_4.7H_2O$	$AlK(SO_4)_2\ 12HO$ $SnO_2+Sr(NO_3)_2\ 4H_2O$ $CaCO_3+ZnSO_4\ 7H_2O$ $Cr_2O_3+MnCl_2+NaCl$

Note.—Best finely powdered, since its colour is then almost destroyed.

For the Preliminary Examination for Acid-radicles (996) :—

Simple.			*Complex.*
$CaCO_3$ $Na_2SO_3\ 7H_2O$ $CaCl_2O$ $NaCl$	FeS KNO_3 $KClO_3$ KI	$Na_2S_2O_3\ 5H_2O$ KBr $Na\bar{A}$	$CaCO_3+KNO_3$ $CaCl_2O+CaF_2$ Na_2CO_3+KI

A few of the above mentioned solids may then be examined by both Preliminary Examinations for Metals and for Acid-radicles.

SUBSTANCES TO BE ANALYSED BY THE GENERAL COURSE IN SECTION VI.

1171. The following lists are so arranged that the analysis of the substances contained in them is more difficult in each column proceeding from left to right, and usually in proceeding in one and the same column from top to bottom. For examples of alkaline solutions, see paragraph 1169.

SUBSTANCES FOR ANALYSIS BY THE GENERAL COURSE.

1. Simple soluble substances.	2. Complex soluble substances.	3. Complex soluble substances.
$BaCl_2.2H_2O$ $MnCl_2$ $HgCl_2$ $MgSO_4.7H_2O$. KCl $NiSO_4.7H_2O$ *$CaCO_3$ $Pb(NO_3)_2$ *As_2O_3 Fe_2Cl_6 NH_4Cl NaCl &c., &c.	$CaCl_2$ $6H_2O+BaCl_2.2H_2O+Sr(NO_3)_2.4H_2O$ $AlK(SO_4)_2$ $12H_2O+CrK(SO_4)_2$ $12H_2O$ $MnCl_2+ZnSO_4$ $7H_2O+CuSO_4.5H_2O$ $Pb(NO_3)_2+HgCl_2+NaCl$ *$As_2O_3+CaCO_3+BaCO_3$ *$ZnO+MgCO_3+KClO_3$ *$MgCO_3+BaCO_3+PbCO_3$ &c., &c.	$\left\{\begin{array}{l}Fe_2Cl_6\\MnCl_2\\ZnSO_4.7H_2O\\KCl\end{array}\right.$ $\left\{\begin{array}{l}As_2O_3\\CrK(SO_4)_2\ 12H_2O\\Co(NO_3)_2.6H_2O\end{array}\right.$ $\left\{\begin{array}{l}Hg_2(NO_3)_2.H_2O\\Sr(NO_3)_2.4H_2O\\KNO_3\\Bi(NO_3)_3\ 5H_2O.\end{array}\right.$ &c., &c.

4. Substances yielding a Phosphate precipitate in Group III.	5. Substances partly or entirely insoluble.	
$\left\{\begin{array}{l}CaCl_2.6H_2O\\Fe_2Cl_6\\Na_2HPO_4\ 12H_2O\end{array}\right.$ $\left\{\begin{array}{l}BaCl_2.2H_2O\\MgCl_2\\Fe_2Cl_6\\Na_2HPO_4.12H_2O\end{array}\right.$ $\left\{\begin{array}{l}Co(NO_3)_2\ 6H_2O\\CaCl_2\ 6H_2O\\MnCl_2\\CrK(SO_4)_2\ 12H_2O\\Na_2HPO_4\ 12H_2O\end{array}\right.$ &c., &c.	$BaSO_4$ SiO_2+S $BaSO_4+CaF_2+SnO_2$ $\left\{\begin{array}{l}BaCl_2.2H_2O\\AlK(SO_4)_2.12H_2O\\AgNO_3\end{array}\right.$ $\left\{\begin{array}{l}SiO_2\\S\\C\end{array}\right.$ $PbSO_4+BaSO_4$ $PbCrO_4$, ignited.	*Metallic substances* (*1067 et seq.*) Iron pyrites (FeS_2) Iron filings (Fe) Zinc clippings (Zn) Brass filings (Cu+Zn) German silver (Cu+Zn+Ni) Bronze (Cu+Sn) Type-metal (Sb+Sn+Pb) *Silicates* (1080)— Fire-clay ($Al_2O_3.2SiO_2$) Brown-clay (do. +Fe) Window-glass (Na, Ca, SiO_2) Flint-glass (Pb, K, SiO_2) *Cyanogen Compounds* (1083)— Any of the foregoing which has been mixed with KCy, KCyS, $K_4FeCy_6.3H_2O$, *$K_6Fe_2Cy_{12}$, $K_6Co_2Cy_{12}$

* To be given in the solid state.

1172. NAMES AND FORMULÆ OF MINERALS CONTAINING COMMON METALS, AND SUITABLE FOR ANALYSIS.

Note.—The principal constituents only are stated below, the traces of impurities or of other constituents are omitted. Minerals which contain the less common elements are not mentioned here, but some of them will be found above the reactions of each of the rarer elements in Section IV. The minerals are ranged under the heading of the principal metal which they contain, and the metals are placed in the order in which they occur in their Groups in Section IV.

Potassium :—

Nitre, or Saltpetre, KNO_3
Potash felspar, or Orthoclase, $K_2O.Al_2O_3\,6SiO_2$, and other double silicates.
Alumstone, $K_2SO_4\,3Al_2(SO_4)(OH)_4$
Silvine, KCl
Kainite (see Mg)
Carnallite, $KCl.MgCl_2.6H_2O$.

Sodium :—

Rocksalt, $NaCl$.
Native soda, $Na_2CO_3\,10H_2O$.
Trona, $Na_2CO_3\,NaHCO_3\,3H_2O$
Cubic nitre, or Chili saltpetre, $NaNO_3$.
Glauber salt, $Na_2SO_4\,10H_2O$
Borax, or Tincal, $Na_2B_4O_7\,10H_2O$
Glauberite, $Na_2SO_4\,CaSO_4$
Cryolite, $AlF_3\,3NaF$
Soda felspar, or Albite, $Na_2O\,Al_2O_3\,6SiO_2$; and other double silicates

Magnesium :—

Periclase, MgO.
Brucite, MgO_2H_2
Magnesite, $MgCO_3$
Dolomite, or Magnesian Limestone, $(Mg,Ca)CO_3$.
Kainite, $MgSO_4\,KCl.6H_2O$.
Kieserite, $MgSO_4.H_2O$.
Epsomite, $MgSO_4.7H_2O$.
Meerschaum, $Mg_2H_2 3SiO_3.H_2O$, and other silicates.
Boracite
Spinel, MgO,Al_2O_3

Barium :—

Heavy-spar, $BaSO_4$.
Witherite, $BaCO$.

Strontium :—

Celestine, $SrSO_4$.
Strontianite, $SrCO$.

Calcium :—

Calcspar, Arragonite, Marble, Chalk, Limestone, $CaCO_3$.
Gypsum, $CaSO_4\,2H_2O$.
Alabaster, Selenite, Anhydrite, $CaSO_4$
Fluorspar, CaF_2
Apatite, $Ca_3(PO\ ).Ca(Cl,F)_2$.

Aluminium :—

Corundum, Al_2O_3.
Bauxite, $Al_2O_3\,H_2O\,(Fe_2O_3)$
Diaspore, $Al_2O_3\,H_2O$.
Alumstone (see Potassium)
Cryolite, $AlF_3\,3NaF$.
Felspar (see K and Na), and other silicates.
Clay, $Al_2Si_2O_7\,2H_2O$.

Iron :—

Meteorite, Fe + Ni
Red hæmatite, Fe_2O_3
Specular iron ore, Fe_2O_3
Brown hæmatite, $Fe_4O_3(OH)$.
Magnetic iron oxide, Fe_3O_4.
Iron pyrites, FeS_2
Spathic iron ore, $FeCO_3$: mingled with clay in Clay-ironstone, and with bitumen in Blackband-ironstone.

Chromium :—

Chrome ironstone, $FeCr_2O_4$.
Crocoisite, $PbCrO_4$.

Zinc :—

Red zinc ore, ZnO.
Zinc blende, ZnS.
Calamine, $ZnCO_3$
Silicious or Electric Calamine, $ZnSiO_4.H_2O$.
Franklinite, $(Zn, Fe)\,O\,Fe_2O_3$

Manganese :—

Pyrolusite, MnO_2
Braunite, Mn_2O_3.

Hausmannite, Mn_3O_4.
Manganite, $Mn_2H_2O_4$.
Psilomelane, (Mn, Ba) $O.MnO_2$
Manganese spar, $MnCO_3$

Nickel :—
Kupfernickel, $NiAs$
Nickel glance, $Ni(As, S)_2$
Nickel silicate, $2NiO,3SiO_3$

Cobalt :—
Speiss Cobalt, $(Co, Ni, Fe)As_2$
Cobalt glance, or Smaltine, $(Co, Fe)(As, S)_2$

Mercury :—
Cinnabar, HgS.

Lead :—
Crocoisite, $PbCrO_4$.
Galena, or Lead spar, PbS
Cerussite, $PbCO_3$
Anglesite, $PbSO_4$
Leadhillite, $PbSO_4\,3PbCO_3$

Bismuth :—
Bismuth ochre, Bi_2O_3.
Bismuthite, or Bismuth glance, Bi_2S_3, and double sulphides.

Copper :—
Red copper ore, Cu_2O.
Copper glance, Cu_2S.
Peacock copper ore, Cu_2S.
Copper pyrites, $CuFeS_2$
Malachite, $CuCO_3\,CuH_2O_2$.

Cadmium :—
Greenockite, CdS.

Arsenic :—
Realgar, As_2S_2
Orpiment, As_2S.
Arsenical nickel, $NiAs_2$; Kupfernickel, $NiAs$; and many other metallic arsenides and arseno-sulphides.

Antimony :—
Stibnite or Grey Antimony, Sb_2S_3, and antimony sulphide combined with other metallic sulphides.
White antimony, Sb_2O_3.
Red antimony ore, $Sb_2O_3.2Sb_2S_3$

Tin :—
Tinstone, SnO_2.
Cassiterite, SnO_2.

Silver :—
Silver glance, Ag_2S
Horn silver, $AgCl$
Pyrargyrite, Ag_3SbS_3
Proustite, Ag_3AsS_3.

Silica :—
Quartz, SiO_2.
Flint, SiO_2.
Rock crystal, SiO_2.

LIST OF CHEMICAL ELEMENTS, WITH THEIR SYMBOLS AND ATOMIC WEIGHTS.

1173 The words in brackets are the Latin names of the elements from which the symbols have been derived. The numbers are the International Atomic Weights for 1907.

Name.	Symbol	Atomic Weight	Name	Symbol	Atomic Weight
Aluminium	Al	27·1	Molybdenum	Mo	96·0
Antimony (Stibium)	Sb	120·2	Neodymium	Nd	143·6
Argon	A	39·9	Neon	Ne	20
Arsenic	As	75·0	Nickel	Ni	58·7
Barium	Ba	137·4	Nitrogen	N	14·01
Beryllium or Glucinum	Be or Gl	9·1	Osmium	Os	191
			Oxygen	O	16·00
Bismuth	Bi	208·0	Palladium	Pd	106·5
Boron	B	11·0	Phosphorus	P	31·0
Bromine	Br	79·96	Platinum	Pt	194·8
Cadmium	Cd	112·4	Potassium (Kalium)	K	39.15
Cæsium	Cs	132·9	Praseodymium	Pr	140·5
Calcium	Ca	40·1	Radium	Rd	225
Carbon	C	12·00	Rhodium	Rh	103·0
Cerium	Ce	140·25	Rubidium	Rb	85·5
Chlorine	Cl	35·45	Ruthenium	Ru	101·7
Chromium	Cr	52·1	Samarium	Sa	150·3
Cobalt	Co	59·0	Scandium	Sc	44·1
Columbium	Cb	94	Selenium	Se	79·2
Copper (Cuprum)	Cu	63·6	Silicon	Si	28·4
Erbium	Er	166	Silver (Argentum)	Ag	107·93
Europium	Eu	152	Sodium (Natrium)	Na	23·05
Fluorine	F	19·0	Strontium	Sr	87·6
Gadolinium	Gd	156	Sulphur	S	32·06
Gallium	Ga	70	Tantalum	Ta	181
Germanium	Ge	72·5	Tellurium	Te	127·6
Gold (Aurum)	Au	197·2	Terbium	Tb	159·2
Helium	He	4·0	Thallium	Tl	204·1
Hydrogen	H	1·008	Thorium	Th	232·5
Indium	In	115	Thulium	Tm	171
Iodine	I	126·97	Tin (Stannum)	Sn	119·0
Iridium	Ir	193·0	Titanium	Ti	48·1
Iron (Ferrum)	Fe	55·9	Tungsten (Wolframium)	W	184
Krypton	Kr	81·8			
Lanthanum	La	138·9	Uranium	U	238·5
Lead (Plumbum)	Pb	206·9	Vanadium	V	51·2
Lithium	Li	7·03	Xenon	Xe	128
Magnesium	Mg	24·36	Ytterbium	Yb	173·0
Manganese	Mn	55·0	Yttrium	Yt	89·0
Mercury (Hydrargyrum)	Hg	200·0	Zinc	Zn	65·4
			Zirconium	Zr	90·6

1174. THERMOMETRIC SCALES.

There are two thermometric scales in use, the Centigrade and the Fahrenheit; the former of these is rapidly becoming universal for scientific purposes. The temperatures, which are occasionally referred to in this treatise, are given on the Centigrade scale. The two scales are mutually convertible by the following formulæ, in which F.° represents a temperature on the Fahrenheit scale, and C.° a temperature on the Centigrade scale:—

$$\text{F.}^\circ = \frac{9\ \text{C.}^\circ}{5} + 32$$

$$\text{C.}^\circ = \frac{5(\text{F.}^\circ - 32)}{9}$$

WEIGHTS AND MEASURES.

1175. The corresponding values of the French and English Weights and Measures are given in the following tables. The use of the French or decimal system is strongly recommended by its extreme simplicity. The smaller denominations are obtained by taking a tenth, hundredth, thousandth, &c., of the unit chosen; and they are designated by the Latin prefixes deci-, centi-, milli-, &c. The higher denominations are 10 times, 100 times, 1000, &c., times the unit, and are named by the Greek prefixes deca-, hecto-, kilo-, &c.

The starting-point of the French system is the "metre" (=39·37 inches); this is the "unit of length." The "unit of measure" is the "litre," which is one cubic decimetre. The "unit of weight" is the "gramme" or "gram," which is the weight of 1 cubic centimetre of distilled water at 4° C. (See *Note* p. 502.)

The chief conveniences arising from the use of this system are:—

1st. That all the different denominations can be written as one; since they are either multiples by ten, or are decimal fractions, of the unit. Thus 5 decagrammes, 3 grammes, 4 decigrammes, 8 milligrammes would be written 53·408 grammes.

2nd. That since 1 cubic centimetre of water at 4° C. weighs 1 gramme, we may obtain the *weight* of a known *measure* of water by simply converting the measure into cubic centimetres, the number thus obtained will represent the corresponding weight of the water in grammes. Of course this conversion is strictly accurate only when the water is measured at 4° C. But for ordinary purposes the error introduced, when the water is at the temperature of the air, is too small to be of any importance in the preparation of solutions.

The weights and measures, which are most frequently used for chemical purposes, are the gramme, the millimetre (mm.), the litre, and the cubic centimetre (c.c.), which is $\frac{1}{1000}$ of a litre.

1176. ENGLISH WEIGHTS AND MEASURES.

Apothecaries Weight.

lb		oz		drms		scruples		grains.
1	=	12	=	96	=	288	=	5760
		1	=	8	=	24	=	480
				1	=	3	=	60
							=	20

Avoirdupois Weight.

lb.		oz.		drms		grains.
1	=	16	=	256	=	7000
		1	=	16	=	437 5
				1	=	27·343

Imperial Measure.

gallon.		pints.		fluid oz		fluid drms.
1	=	8	=	160	=	1280
		1	=	20	=	160
				1	=	8

1 gallon = 70,000 grains of water at 16°·7 C.
1 fluid ounce = $\frac{1}{20}$ pint = 437·5 ,, ,, ,,
1 gallon = 277 280 cubic inches
1 fluid ounce = 1·733 ,,

1177. FRENCH WEIGHTS AND MEASURES.

MEASURES OF LENGTH. ENGLISH.

	metre.		inches.		mile	furlong	yards	feet.	inches
Millimetre =	0 001	=	0 03937	=	.	.	.	.	0 03937
Centimetre =	0 01	=	0 39371	=	.	.	.	.	0 39371
Decimetre =	0 1	=	3 93708	=	.	.	.	.	3 9371
Metre =	1 0	=	39 37079	=	.	.	.	3	3 371
Decametre =	10 0	=	393 70790	=	.	.	10	2	9 7
Hectometre =	100 0	=	3937 07900	=	.	.	109	1	1
Kilometre =	1000 0	=	39370 79000	=	.	4	213	4	10 2
Myriometre =	10000 0	=	393707 90000	=	6	1	156	0	6

1 inch = 0 0254 metre = 2 5399 centimetres.
1 foot = 0 3048 ,,
1 sq. inch = 6·4514 sq. centimetres.

MEASURES OF CAPACITY.

1 litre = 1 cubic decimetre.

		litre.		cubic inches.		pints.
Millilitre, or Cubic centimetre (c c)	=	0·001	=	0·06103	=	0·00176
Centilitre	=	0·01	=	0·61027	=	0·01761
Decilitre	=	0·1	=	6·1027	=	0·17608
Litre	=	1·0	=	61 027	=	1·76077
Decalitre	=	10·0	=	610·27	=	17·60773
Hectolitre	=	100 0	=	6102 7	=	176·07734
Kilolitre	=	1000 0	=	61027 0	=	1760·77341
Myriolitre	=	10000·0	=	610270 0	=	17607·73414

1 cubic inch = 0·01639 litre.
1 cubic foot = 28·31531 litres.
1 gallon = 4·54336 ,,

MEASURES OF WEIGHT.

1 gram = the weight of 1 cubic centimetre (c.c.) of water at 4° C.

		grams.		grains.	Avoirdupois.			
Milligram	=	0·001	=	0·01543				
Centigram	=	0·01	=	0 15432				
Decigram	=	0·1	=	1 54323				
Gram	=	1 0	=	15·43235		lbs.	oz.	drms.
Decagram	=	10 0	=	154·32349	=	0	0	5 65
Hectogram	=	100 0	=	1543·23488	=	0	3	8 5
Kilogram	=	1000 0	=	15432·34880	=	2	3	5
Myriogram	=	10000 0	=	154323·48800	=	22	1	2

1 grain = 0 0649 gram.
1 oz. (Troy) = 31 1035 grams.
1 lb. (Avoirdupois) = 453·593 ,,

Note —In curtailing the word from *gramme* to *gram*, the risk of mistaking *gram* for *grain* or *vice versâ* must be guarded against, as such a mistake would lead to very serious error

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